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JOURNAL

OF THE

AMERICAN CHEMICAL SOCIETY.

VOLUME IV.

PUBLICATION COMMITTEE

ELWYN WALLER,
CHARLES A. DOREMUS,
L. H. FRIEDBURG.

ABSTRACTORS

A. H. ELLIOTT, L. H. FRIEDBURG,
A. BOURGOUGNON, O. H. KRAUSE,
PERCY NEYMANN.

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TABLE OF CONTENTS.

VOLUME IV.

Proceedings, January 6th, 1882 1
I.—Upon the Reduction of Metallic Oxides in Sunlight. By ALBERT R. LEEDS, Ph.D. 3
II.—On the Estimation of Tannic Acid in Tanning Materials, etc. By NELSON H. DARTON 4
III.—On a Convenient and Accurate Form of Weighing Flask to Deliver as a Burette. By NELSON H. DARTON 6
Obituary of Dr. Joseph Goldmark 7
Proceedings, February 3d, 1882 10
IV.—On Crystallized Anhydrous Grape Sugar. ARNO BEHR, Ph.D. 11
V.—The Water Supply of the City of New York. By ELWYN WALLER, Ph.D. 15
VI.—Heptylene from Heptane of P. Sabiniana. By F. P. VENABLE, Ph.D. 22
Proceedings, March 3d, 1882 24
VII.—Determination of Zinc as Pyrophosphate. By GEORGE C. STONE, Ph.D. 26
VIII.—Methyl Violet Test Paper. By T. O'CONNOR SLOANE, A.M., Ph.D. 31
IX.—Diphenylamine-Acrolein. By ALBERT R. LEEDS, Ph.D. 32
Proceedings, April 7th, 1882 35
X.—Volumetric Estimation of Copper and of Lead. By P. CASAMAJOR 35
Foreign Patents. Condensed from R. Biedermann's Report to the German Chemical Society, by O. H. KRAUSE 38
XI.—On the Comparative Value of the Several Methods of Determining Tannic Acid. By NELSON H. DARTON 49
Proceedings, May 5th, 1882 57
XII.—Acrolein—Urea. By ALBERT R. LEEDS, Ph.D 58
XIII.—Note on an Earthy Ferric Sulphate from Arkansas. By CHARLES E. WAIT 61
XIV.—On the Comparative Value of the Several Methods of Determining Tannic Acid. Part II. By NELSON H. DARTON 62
Review of Industrial Chemistry. By A. A. BRENNEMAN 72
Abstracts. Comptes Rendus. By A. BOURGOUGNON 76
Foreign Patents. By O. H. KRAUSE 78
Upon the Preparation of Artificial Indigo and some of its Derivatives. By JAS. H. STEBBINS, JR., S.B. 81
Proceedings, Adjourned Meeting, May 19th, 1882 87
XV.—An Examination of some of the Methods of Determining Phosphorus in Iron Ores. By ELWYN WALLER, Ph.D. 88
Proceedings, June 3d, 1882 92
XVI.—Acetate of Lime; its Manufacture and Analysis. By C. M. STILLWELL and T. S. GLADDING 94
Foreign Patents. O. H. KRAUSE 108
XVII.—On the Determination of Reverted Phosphates. By THOMAS S. GLADDING, A.M. 113
XVIII.—On Contamination of the New York Water Supply. By ALBERT R. LEEDS, Ph.D. 127
XIX.—On the Estimation of Phosphoric Acid as Magnesic Pyrophosphate. By THOMAS S. GLADDING, A.M. 135
Abstracts. Journal of the Chemical Society, London. By A. H. ELLIOTT, Ph.B., F.C.S. 141

36529
9479

XX.—On the Properties of Pure Metallic Aluminium. By. J. W. MALLET, F.R.S..... 145
XXI.—Contributions from the Laboratories of the School of Mines, Columbia College. On Nitro-saccharose. By ARTHUR H. ELLIOTT, Ph.B., F.C.S..... 147
XXII.—Notes on Bone Oil. By ARTHUR H. ELLIOTT, Ph.B., F.C.S., and FERDINAND SANDS, A.B., Ph.B..... 153
XXIII.—On the Composition of Elephants' Milk. By CHARLES A. DOREMUS, M.D., Ph.D. 157
Review of Papers on Analytical Chemistry (Inorganic) which have appeared during the year 1882. By E. WALLER, Ph.D. 160
Abstracts. Journal of the Chemical Society, London. By A. H. ELLIOTT..... 168
Berichte der Deutschen Chemischen Gesellschaft. By L. H. FRIEDBURG, Ph.D. 173
XXIV.—On the Estimation of Sulphuretted Hydrogen in Gas by Direct Weight. By A. P. HALLOCK..... 177
XXV.—On the Fractional Dehydration of Ammonium Alum, and the Atomicity of Aluminium. By. J. W. MALLET, F.R.S..... 180
XXVI.—On Nitro-saccharose. A correction. By A. H. ELLIOTT, Ph.B., F.C.S..... 186
Review of Papers on Analytical Chemistry (Proximate) which have appeared during the year 1882. By E. WALLER, Ph.D..... 188
Abstracts. Journal of the Chemical Society, London. By A. H. ELLIOTT 198
Berichte der Deutschen Chemischen Gesellschaft. By L. H. FRIEDBURG..... 204
Comptes Rendus. By A. BOURGOUGNON..... 208
Proceedings, October 6th, 1882..... 211
XXVII.—Laboratory Notes. By ELWYN WALLER, Ph.D..... 212
XXVIII.—Laboratory Notes. By JAS. H. STEBBINS, Jr., Ph.B., F.C.S..... 214
XXIX.—Analyses of the Calculi in the "Wood" Museum of Bellevue Hospital. By A. BOURGOUGNON 215
Abstracts. Journal of the Chemical Society, London. By A. H. ELLIOTT..... 219
Berichte der Deutschen Chemischen Gesellschaft. By PERCY NEYMANN..... 226
Comptes Rendus. By A. BOURGOUGNON..... 230
Proceedings, November 3d, 1882..... 243
XXX.—On the Action of Phthalic Anhydride upon Gallic Acid. By JAS. H. STEBBINS, Jr., A.B., F.C.S..... 244
XXXI.—Note on Asbestos Filters. By A. CASAMAJOR..... 248
XXXII.—Fat Extracting Apparatus. By T. O'CONNOR SLOANE, Ph.D..... 250
XXXIII.—On Bi-sulphide of Carbon. By L. H. FRIEDBURG, Ph.D..... 252
XXXIV.—On the Action of Alcoholic Potash on Heptylene Bromide. By F. P. VENABLE, Ph.D..... 254
XXXV.—Analysis of Rock Salt from Saltville, Washington County, Va. By THOMAS RADCLIFFE..... 255
XXXVI.—Analysis of a Deposit of Zinc Oxide in a Blast Furnace at Longdale, Va. By THOMAS RADCLIFFE..... 256
Abstracts. Journal of the Chemical Society, London. By A. H. ELLIOTT..... 257
Berichte der Deutschen Chemischen Gesellschaft, Berlin. By PERCY NEYMANN. 261
Comptes Rendus. By A. BOURGOUNON..... 272
Proceedings of December 8th, 1882 290
XXXVII.—On the Manufacture of Tartaric Acid. By L. H. FRIEDBURG, Ph.D. 292
XXXVIII.—On the Action of Phthalic Anhydride upon Gallic Acid. By GEO. A. PROCHAZKA. 296

PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

Meeting held January 6th, 1882.

The meeting was called to order at 8:20 p. m., Dr. A. R. Leeds in the chair.

The minutes of the previous meeting were read and approved.

The report of the board of directors was read and approved.

After which Mr. Elliott stated that he attended Mr. John Lipps' funeral together with Dr. Waller and some other gentlemen, and that the expenses incurred amounted to about $116, of which he had only raised $96.50 by subscription, the balance having been paid out of his own pocket.

The recording secretary then moved that Mr. Elliott be reimbursed from the society's funds.

It was then amended, that Mr. Elliott's claim be brought up at the next meeting of the board of directors for consideration.

This on motion was carried.

After which the following gentlemen were duly elected members of the society:

Clement Le Boutillier, associate member, proposed by A. H. Elliott, T. O'C. Sloane, A. R. Leeds.

Victor M. Braschi, regular member, proposed by A. H. Elliott, T. O'C. Sloane, A. R. Leeds.

F. G. Wiechman, regular member, proposed by A. H. Elliott, T. O'C. Sloane, A. R. Leeds.

Lucius Pitkin, regular member, proposed by A. H. Elliott, T. O'C. Sloane, A. R. Leeds.

Charles A. Wittmack, associate member, proposed by A. H. Elliott, T. O'C. Sloane, A. R. Leeds.

Dr. Henry Rau, regular member, proposed by James H. Stebbins, Jr., O. M. Jenks, A. R. Leeds.

Dr. J. A. Peters, regular member, proposed by S. A. Goldschmidt, C. C. Parsons, James H. Stebbins, Jr.

Dr. J. Rudesch, regular member, proposed by M. Alsberg, James H. Stebbins, Jr., H. Endemann.

After which Mr. Orazio Lugo was nominated as a new member.

Dr. Endemann then stated, in behalf of the committee on papers

and publications, that he did not expect the next number of the Journal for 1881 would be out before February 1st, 1882.

After which the first paper of the evening, "Upon the influence of light, upon certain metallic oxides," by Dr. A. R. Leeds, was read.

After some questions by Mr. Elliott, the second paper, "On the estimation of tannic acid, in tanning materials," by Mr. Nelson H. Darton, followed.

This paper caused a very lively discussion, between some of the gentlemen present.

After which the third paper, "On a convenient and accurate form for a weighing flask, to deliver as a burette," by Mr. Nelson H. Darton, was read.

After some remarks by Dr. Grothe and others, Mr. Elliott announced the death of Dr. John W. Draper, and moved that a committee of two be appointed, to wait on Prof. Chandler, and request him to draw up an obituary notice. This being seconded was duly carried.

Dr. Friedburg then described a new test paper, manufactured by Toch, Grothe & Co., which has the property of distinguishing between mineral and organic acids.

This paper has a blue shade, and is not acted upon by organic acids.

Dr. Friedburg then proposed that our Conversaziones should be made more interesting, and urged that some paper or theme should be settled upon beforehand, the title of which should be sent to the recording secretary, to be announced on the postal cards.

This being seconded, was duly carried.

After which the recording secretary stated that he would give a paper on artificial indigo, with samples, for the next Conversazione.

Mr. Casamajor then announced the resignation of Mr. W. B. Hills and Chas. F. Wingate, both of which were duly accepted.

Mr. Elliott then asked whether any action had been taken about members that are in arrears of dues.

This being answered in the negative, Mr. Casamajor then moved that all members in arrears of dues up to Dec. 31, 1880, be dropped from the rolls.

This being seconded, was carried unanimously. After which the meeting adjourned.

JAMES H. STEBBINS, JR.,
Recording Secretary.

I. UPON THE REDUCTION OF METALLIC OXIDES IN SUNLIGHT.

By Dr. Albert R. Leeds.

During the course of some experiments upon the measurement of solar actinism, I was led to subject certain metallic oxides to the influence of sun-light during a very prolonged period and under somewhat novel conditions. The oxides were contained in W-shaped tubes, from which the air was displaced by a current of pure and dry hydrogen continued during five hours. After exhausting the atmosphere of hydrogen as completely as possible, the tubes were sealed. The oxides were placed in one bend of the tube, and in the other bend metallic sodium, or, in some experiments, anhydrous cupric sulphate. All of the tubes were filled upon the 10th of November, 1880, and exposed at a southern window until Nov. 10th, 1881, when they were examined and opened.

I. A tube with 5.2256 grms. mercurous oxide in one bend, and sodium in the other. The oxide was largely reduced to metallic mercury, which formed a mirror with globules, and to *yellow* mercuric oxide. The sodium was covered with a white crust. On account of the mingling of the globules of mercury with the reduced and unreduced mercurous oxide, no quantitative estimations could be effected.

II. A V-tube containing 5.4064 grms. mercurous oxide, but no sodium. A mercury mirror was formed, and the residue consisted of a mixture of mercurous oxide and *yellow* mercuric oxide. As in the preceding experiment no red oxide of mercury was found.

The two preceding experiments are at variance with the results of Dulk, quoted in Gmelin-Krauts Handb. der Anorg. Chemie. Vol. I, p. 825, according to which the mercurous oxide was resolved into mercury and *red* oxide of mercury.

III. A W-tube containing 6.3597 grms. red oxide of mercury in one bend and metallic sodium in the other. No decomposition ensued, the weight remaining unaltered. This result likewise differs from that of Dulk (loc. cit.), who found that dry mercuric oxide lost in 4 months under colorless glass 0.9, under violet 0.5, under green 0.2 and under red 0.1 per cent. of oxygen. At the same time its color changed to gray, most strikingly under colorless, but scarcely perceptibly under red glass.

IV. A W-tube containing 3.043 grms. of pure PbO_2 in one bend

and metallic sodium in the other. The weight and appearance of the peroxide remained unaltered.

This result differs from that of Dulk (loc. cit.), the brown oxide of lead being decomposed into oxygen and the red oxide.

V. 4.057 grms. of PbO_2 was used in this experiment, but instead of sodium, anhydrous cupric sulphate was placed in the other bend of the tube. Neither the lead peroxide nor the copper salt changed in appearance, nor the former in weight.

II. ON THE ESTIMATION OF TANNIC ACID IN TANNING MATERIALS, ETC.

By Nelson H. Darton.

As the exact determination of the proportions of tannic acid is a problem of comparatively difficult solution many methods have been proposed to facilitate this, but, as a rule, have yielded very discordant results when compared with the actual weights of the leather formed from it. Thus tanners have lost all faith in chemists generally. Some prominent tanners have informed me that they have sent spent barks containing less than one per cent of tannic acid to chemists in this city and they have returned results of from 7 per cent. to 4 per cent. These results were generally obtained by Hammels hide method or that of Lœwenthal, as modified by Neubauer, or even by the old gelatin method, which is now seldom used.

In my intimate connections with the tanning trade I seriously felt the defects in and want of a proper method, and not being able to find one of sufficient accuracy sought out one that would answer the conditions of speed and accuracy, and this I will detail below, merely mentioning that in numerous and exhaustive trials I have found it to yield results as near as within $\frac{1}{4}$ per cent of the actual value. Lœwenthal's modification of his own method is not nearly as accurate as this, as I will show in a subsequent paper.

The bark, etc., is taken dry and finely cut, powdered or crushed. 20 grammes of this is weighed out, placed in a flask with sufficient water and the tannic acid thoroughly exhausted by repeated treatments with boiling water. The filtrate is mixed with about 25 cc. of dilute sulphuric acid and again filtered after making the bulk up to one litre. Standard solutions are then prepared. The first of tannic acid containing two grammes to a litre. The pure tannic acid must be used. 2nd, a solution contain-

ing to a litre 1.5 grammes of permanganate of potassium, 3rd, a solution of indigo carmine of such a strength that it must require twice as much of the permanganate solution as an equal bulk of the tannin solution, that is about 3 grammes to a litre. 4th, a 10 per cent solution of ammonio-sulphate of copper in ammoniacal water. 5th, a dilute sulphuric acid, one of acid to five of water. A convenient measure of the indigo is taken, say 20cc. It is diluted to about $\frac{3}{4}$ of a litre, acidulated with the acid and titrated with the permanganate. Another amount of the indigo and the same of tannic acid solution are taken and titrated as before. This is then repeated, using the decoction of the bark. About 60 cc. of the last is then taken and about the same of the copper solution, these are mixed together, some ammonia solution added and the mixture filtered from the tannate of copper which precipitates. An equivalent amount of this filtrate is titrated as before and from these data the amount of tannic acid calculated by the following formula.

The amount of permanganate used for the indigo and tannin together, less that used by the indigo, is to the permanganate used for the decoction, less that necessary for the indigo present and an equivalent amount of the filtrate noted, as the amount of tannic acid present in the litre of standard solution, is to the quantity of tannic acid in 20 grammes of the bark (a litre of decoction). The process may be executed in abont one half hour after the decoction is obtained, but as eight or ten of the determinations may be carried on at once without loss of time it is by far the shortest method known.

This process has been in constant use in my laboratory for some time back, and as I have much of this work constantly to attend to I have thoroughly tested its value and am assured the main error is not over $\frac{1}{4}$ per cent. as I will show in my subsequent paper.

This method, somewhat extended, is applicable to the determination of the gallic acid at the same time. It is simply in treating a portion of the filtrate from the tannate of copper with a solution of bichloride of mercury, and after filtering this off and separating the excess of Hg, titrating the filtrate, using the calculations similar to the above, the amount of gallic acid may be readily arrived at. The relation of the permanganate to a gallic acid solution for the first member of the equation should be previously ascertained, although it differs but slightly from the tannic acid.

I then proceeded to investigate the purity and properties of the tannate of copper upon which the whole of the method depends. Two tannates of copper were prepared, one from the pure

tannic acid, the other from the decoction of hemlock bark and the two were compared, the first, from the pure tannic acid, contained 27.72 per cent. of copper oxide and the other 27.58 per cent., within 99.5 per cent. of each other. By combustion they both yielded essentially the same formula. In both cases they were in black lustrous masses quite insoluble in cold but partly soluble in hot or boiling water, from which it is deposited on cooling. In alcohol, gallic acid solutions and in presence of alkalies they are very insoluble but quite soluble in diluted acids.

III. ON A CONVENIENT AND ACCURATE FORM OF WEIGHING FLASK TO DELIVER AS A BURETTE.

By Nelson H. Darton.

In many volumetric processes, the end reaction is so sharp that the small amount of solution required to produce it is too small to be perceptible in the reading of an ordinary burette. Readings to 0.05 cc. are as close as can be usually seen. Differences of temperature, which affect both the bulk of the solution and the capacity of the burette, are also sources of error which must be avoided if we wish to attain great precision.

In cases where extreme accuracy is required, the method of weighing, instead of measuring the amount of solution used, is to be preferred.

Two or three forms of apparatus for this purpose have been proposed, but they all are open to two objections. 1st. That the delivery tube is too large to deliver less than 0.05 cc., or if fine enough to deliver it, a sufficient amount cannot be delivered conveniently at the start to bring the solution upon which one is working near to the point of change, and 2d. that regulating the admission of the air to the apparatus distracts the operator's attention at the critical moment.

I have constructed and used an apparatus which is not open to the above objections. Two forms are used. The first for liquids, which may suffer an alteration of standard by contact with organic substance, as cork or rubber, consists of a flask with a Gay-Lussac delivery tube with a fine delivery jet. The stopper is perforated for a glass tube, connecting by a short rubber tube with another tube drawn to a fine point to regulate the admission of air to the flask. A small pinch cock on the rubber tube completes the control of the air supply, and consequently of the delivery from the flask. With the

apparatus I use three or more of these tubes for regulating the air supply, which can be weighed with the flask and changed as required.

The second form of apparatus is essentially the same, except that the solution is delivered from the flask by a small siphon passing through the cork, the supply being regulated as before.

OBITUARY.

JOSEPH GOLDMARK.

From the small band of men by whom the project of an American Chemical Society was agitated, we have lost one who entered into it with enthusiasm and who, ever since the founding of the society, has rejoiced in its existence—Dr. Joseph Goldmark.

Although born and educated abroad and strongly attached to the land of his birth, the active part of Dr. Goldmark's life was passed in the city of New York, where he built up his large manufacturing business and where all his interests were centered.

He was born in the small town of Kreuz in Hungary.

At the age of seventeen he entered the University of Vienna, where he took his degree of Doctor of Medicine. At the same time he devoted himself to the study of chemistry with great zeal, working in the laboratory under Prof. Schroetter. Here he applied himself to the investigation of amorphous phosphorus, many of the remarkable properties of which he claims to have discovered. It was his intention to assert his claim to these discoveries in the scientific journals, and on the occasion of his last visit to Europe he was engaged in collecting material to establish his title to them.

In the revolution of 1848, which brought the first dawn of political liberty to Austria, Joseph Goldmark took part as a leader. In connection with such men as Fischhof, Violand, Füster and others he will be remembered as the bold and enthusiastic advocate of reform.

In the stormy days of March, 1848 he was at the head of the Legion of Students who left their lecture hall, opposed the professors, and fifteen hundred strong took possession of the Chamber of Deputies and forced the "Staende" to petition the emperor for

reforms, including liberty of the press, religious liberty, representation by the people, etc.

He was chosen as one of the twelve delegates sent by the students to the imperial palace, and took part in all the stirring measures of that memorable period.

But the glorious early days of the movement, so full of high aspirations and of enthusiasm for liberty, were followed by darker days, when the spirit of loyalty and moderation was supplanted by the blind fury of the mob.

It was in such days that the names of the exalted leaders became connected with the deeds of violence which they themselves held in abhorrence.

Thus when Latour, the hated Minister of War, fell (Oct. 6, 1848) slain by the populace, Goldmark and Fischhof were the two delegates who sought to save him, endangering their own lives to save the minister's.

When the brief reign of the multidude was over, and absolutism again set in, the leaders of the revolution were held to account for the wild deeds of the mob, and an unjust retribution fell upon innocent men.

Fischhof lingered long in prison, while Goldmark escaped across the border. But, indicted as the instigators of the murder of Latour, they were condemned to death on the testimony of one witness and the sentence was carried out in effigy.

Goldmark and other fugitives reached Switzerland in safety and thence proceeded to the United States in 1849.

After one year's practice of his profession, Dr. Goldmark abandoned it for the more congenial pursuit of experiment and manufacture. For some time he was engaged in experiments with fulminating powders, the manufacture of which he reduced to a comparatively safe process, and in 1858 he established in Brooklyn a a factory for cartridges and percussion caps.

To this work he gave the best years of his life, personally superintending the manufacture, inventing machinery, patenting improvements and competing with foreign manufacturers, his chemical knowledge being seconded by considerable mechanical ingenuity.

In 1868 he returned once more to his native Austria, not to avail himself of the amnesty which the emperor had extended to the banished revolutionists, but in order to secure a just trial and to prove his innocence.

He returned—on the statute books still the condemned murderer —but, a trial being granted, he was triumphantly acquitted, overwhelming proof showing that the witness whose testimony had convicted him had been bribed to swear to false statements. And not only was the innocence of Dr. Goldmark thus fully demonstrated, but it was also made apparent in what regard the people still held the men of '48; for at the close of the trial a public ovation was tendered to him,in which all the students and professors of the university joined as well as many eminent men in public stations.

Returning to his adopted country Dr. Goldmark continued his quiet life as a private citizen, finding leisure in spite of numerous business cares for constant attention to the interests of the political situation as well as for scientific pursuits. Although he never held an office he devoted much of his time and means to the service of political reform, holding it to be the duty of every true man to give a part of his attention to public matters, discouraging though the field of party strife may be.

The responsibilities and cares connected with the management of his large business left him no time in the latter years of his life for the prosecution of original research in the domain of pure chemistry; but the part he took in the discussions at the meetings of the society and his conversation showed how well he had kept pace with the progress of science.

Repeatedly elected a member of the Board of Directors and chosen to serve on various committees, Dr. Goldmark devoted much of his time to the business of the society, giving it the benefit of his wide experience and sound judgment.

Upright, just, accomplished and of a genial disposition he was loved by those who enjoyed his friendship and respected by all who knew him. By his death the society loses a distinguished member and the community an exemplary citizen. He belonged to that class of men whose watchward is: "the best and highest only," and his lifework was thorough, reliable and self-forgetful.

He died at the age of sixty-two on April 18th 1881.

O. H. KRAUSE.

PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

Room 1, *University Building, February* 3, 1882.

The meeting was called to order at 8.20 P. M., Prof. A. R. Leeds in the chair.

The minutes of the previous meeting were read and approved.

The report of the Board of Directors was then read.

The Librarian then stated that he had received the missing numbers of the Berichte der D. Chem. Gesell. along with a number of other pamphlets, etc.

The Librarian on being asked whether he had taken any steps in regard to the subject brought up at the last meeting of the Board of Directors, stated that he had had a number of cards printed which he would furnish on application to any member desiring to use the library.

He furthermore stated that the janitor had consented to take down the names of all gentlemen desirous of using the library.

Dr. Geyer then stated that the $19.50 due to Mr. Elliott for expenses incurred on Dr. J. Lipps' funeral had been collected and paid.

The Chairman of the Committee on Papers and Publications had no report.

The question of dropping members in arrears of dues was then brought up, and ended in the following motions by Mr. Casamajor.

1. That all members in arrears of dues to Dec. 31, 1879, be dropped from the rolls.

This, on being seconded, was carried unanimously.

2. That a committee, consisting of the Treasurer and the two Secretaries, be appointed to draw up a list of members whose dues have been properly paid. Carried.

Mr. Elliott then made some remarks about the publishing of the next number of the Society's Journal, and finally stated that at the next meeting of the Society, he would move an amendment to the Constitution relating to the Committee on Papers and Publications which he wishes to be increased to five.

Mr. Orazio Lugo was then elected as a regular member.

After which, Dr. Emil Hirshberg was nominated as a new member.

The first paper of the evening "On Crystallized Anhydrous Grape Sugar," by Dr. Arno Behr, was then read.

Some remarks by Dr. Waller then followed, after which the second paper "On the precipitation of tannic acid as tannate of copper," by Mr. Nelson H. Darton, was read.

After some remarks and questions by Dr. Grothe the third paper, "On the water supply of N. Y. City," by Dr. E. Waller, was read.

This paper provoked a lively discussion between Drs. Endemann & Waller.

Dr. Endemann then read a paper by title "On Heptane," by F. P. Venable, Ph. D.

After which the meeting adjourned.

JAMES H. STEBBINS, JR.,
Recording Secretary.

IV. ON CRYSTALLIZED ANHYDROUS GRAPE SUGAR.

DR. ARNO BEHR.

The general physical and chemical properties of crystallized anhydrous grape sugar are pretty well known, and I am only able in a few points to supplement this knowledge on this occasion. Anhydrous grape sugar in a state of purity has so far only been obtained from an alcoholic solution. Two years ago F. Soxhlet found that the best solvent for it is methylic alcohol, from which much larger and better developed crystals can be obtained than from the solution in ethylic alcohol. I have found that it can, even more easily, be prepared from a watery solution.

The principle that a crystal introduced into the supersaturated solution of the same substance induces crystallization, has long been applied to the practice of grape sugar manufacture. In order to hasten the hardening of the sugar a certain quantity of the already hardened sugar of a previous operation is stirred into the mass. But as the ordinary commercial grape sugar always contains the hydrate the crystallization so obtained is also that of the hydrate. I put the question to myself, what would happen if, instead of the hydrate, I introduced the anhydrous sugar into a concentrated solution of ordinary grape sugar. I tried the experiment and must confess that I had not much hope that anything else but crystallized hydrate would be the result, for I expected to see the anhydride transformed into the hydrate within the watery solution. I was agreeably surprised when, on the next morning, I found the glass filled with a neat crystallization of anhydrous grape sugar,

from which the liquid part could be easily drained. The few crystals of anhydride, far from being transformed into the hydrate, had induced an ample cyrstallization of their kind. The explanation of this fact is found in the following : In its crystalline form anhydrous grape sugar is not deliquescent even in very moist weather, and it is staple in comparatively dilute solutions of grape sugar. I have kept crystals exposed to the atmosphere of the laboratory through months and during moist weather without seeing them lose their sharp outlines and bright appearance, and I have repeatedly found the syrup drained from a crystallization of anhydrous sugar to contain as much as 26% of water.

The limits of concentration within which this crystallization can be obtained are rather wide, but in order to secure a good result the solution ought to contain from 12 to 13% of water. It is well not to allow the mass to cool rapidly or the temperature to fall much below 30° C. For, at a lower temperature, and before the remaining syrup has been diluted by the separation of the anhydrous crystals, concentrated solutions are rather viscous and this viscosity prevents a free crystallization. A good temperature is 30 to 40° C. The time within which the crystallization is completed varies between half a day and several weeks, according to the purity of the mass.

Though it is always well, in order to secure a uniform and speedy crystallization, to start it by the introduction of some crystals, yet it is possible and, for sugars of high purity, quite easy to obtain the same crystallization by simply keeping the concentrated solutions at a temperature of about 30° C for some time. Under these circumstances a crystallization of anhydrous grape sugar takes place. This behavior of grape sugar is also unexpected. Soxhlet, who, a short time ago, took patents in different countries for the refining of grape sugar by means of alcoholic liquids, and for the production of a hard crystallized grape sugar, describes one of his products expressly as the hydrate of the formula $C_6H_{12}O_6H_2O$, yet he concentrates highly a solution of very pure grape sugar and allows it to crystallize at an elevated temperature. I have failed, under the conditions of my experiments, to obtain the hydrate. But that it is possible for the hydrate to crystallize in large and well developed crystals has been established in 1877 by Halse and Steiner, who analyzed a crystallized hydrate of grape sugar, of which some crystals weighed 4 to 5 grams, and which was readily taken for cane sugar. This grape sugar had made the voyage from England to Australia

and back and during this time had undergone the transformation.

A product which has for some time played an important part in the literature of this subject is Anthon's hard crystallized grape-sugar. As early as 1857, Anthon in Prague prepared a very pure sugar by crystallizing and pressing the hydrate; he then melted the press-cakes without addition of water, and allowed the mass to solidify in moulds.

He obtained crystalline masses which, according to his analysis, contained 4.7% of water and for which he claimed the constitution of a half hydrate of grape sugar of the formula, $2(C_6H_{12}O_6)+H_2O$. As he did not drain his crystals, he certainly had nothing but a mixture of anhydrous sugar and the hydrate, the surplus water of the hydrate having been evaporated during the melting. This has already been suggested by Stohmann in the latest German edition of Muspratt's chemistry (VI 2077).

Crystallized anhydrous grape sugar such as I have prepared it from a watery solution has the following properties. Dried at 30 to 40° C. it does not retain more than .02 per cent. of moisture; the moisture determination made at 130° C. It shows a neutral reaction, with sensitive litmus paper. It melts in a capillary tube between 141 and 145° C. It was tested in the polariscope, and showed birotation. Landolt in his book on the optical rotatory power of organic substances (Braunschweig 1879, p. 184), gives 32.68 grams as the amount of pure grape sugar, which taken instead of the normal weight of cane sugar should show 100 on the scale of a Ventzke-Soleil instrument.

Mr. Lungwitz, who has made these determinations for me, found, if he rapidly dissolved this amount in cold water and immediately polarized the solution, a polarization varying between 202 and 204; if he allowed it to stand for 24 hours, 101 to 102.

This difference is mainly due to an error in Landolt's figure. This figure is calculated from an assumed specific rotation of aD= 53.0. This is correct only for a concentration of 10 grams of sugar in 100 cc. of solution, but for a concentration of 32.68 grams in 100 cc. aD becomes=53.57 according to Tollen's determinations. Therefore 32.68 grams ought to polarize 101.1; while the observed polarization for monorotation was 101 to 102.

These are the facts so far as they refer to chemistry, but in view of the increasing importance of grape sugar as an article of general consumption, I wish to add a few remarks with reference to the industrical application of these observations.

In the ordinary process of the manufacture of grape sugar from starch the conditions are such that the resulting product is always far from being pure grape sugar, however pure the starch from which it is derived may have been. Though a good method for the quantitative determination of starch consists in its conversion with a mineral acid and subsequent determination with Fehling's solution, yet, in practice, a smooth and complete conversion is not attainable. The reason of this difference lies in the fact that the chemist, for a complete conversion, works with a very diluted solution, while the manufacturer necessarily works with solutions of higher density. At a higher density, however, the acid seems to act on the sugar already formed, and before all the dextrine is converted into sugar, the sugar itself is partially converted into something else which constitutes an impurity in the final product. So far we know very little about the nature of these impurities of commercial grape sugar, but several chemists have asserted that the residues which remain after fermentation and distillation are more or less injurious to the human system. This subject, though, requires a more complete investigation. As the principal use of all the grape sugar produced is that which is made of it in the manufacture of fermented beverages, beer and wine, it is easy to understand the rising demand for a purer article.

Fr. Anthon has, twenty years ago, called attention to the disadvantages arising from the use of impure grape sugar in wine making, and suggested a remedy. His suggestion was to refine the ordinary grape sugar by crystallization and the use of a centrifugal machine for the removal of the liquid impurities. He modified this process in so far as he used a strong press instead of a centrifugal machine, and, according to the testimony of several chemists, really produced an article of remarkable purity. His process seems to have never been used for any length of time or on an extensive scale.

Fouchard had already, in 1853, manufactured a refined grape sugar by allowing grape sugar solutions to crystallize in barrels and then withdrawing the liquid portion through a number of holes in the bottoms of the barrels.

Though the principle of these refining processes is correct, yet there is a difficulty inherent in it which arises from the form and nature of the crystals in which the sugar solidifies. Under ordinary circumstances grape sugar crystallizes from a watery solution as the hydrate in the shape of very fine tablets which are mostly

grouped spherically. Owing to the fineness of the tablets and the capillary attraction, it is difficult to remove the impure mother-liquor sufficiently from the crystals by means of a centrifugal machine, and even with a hydraulic press high purity cannot be obtained, together with a large yield. It is different with the crystals of anhydrous grape sugar. They are of a prismatic shape, and form loose aggregations from which the syrup can be easily removed by centrifugal force, and which lend themselves to a treatment of draining and washing very similar to that of cane sugar. Under these circumstances it is possible to produce a grape sugar which compares in purity with block and granulated cane sugar. A number of applications for such an article readily suggest themselves. The confectioner, the druggist, the manufacturer of condensed milk may use it. In the preparation of certain wines it can safely take the place of cane sugar; but its principal use ought to be in the kitchen for all those preparations where utmost sweetness is not sought for. It is not so well suited for sweetening tea or coffee, though it does not quite so unfavorably compare with cane sugar as the books will have it. To obtain a moderate sweetness, equal to that produced by a given amount of cane sugar, it is not necessary to take $2\frac{1}{2}$ or 3 times as much as cane sugar, but only about $1\frac{2}{3}$ times the quantity; at least I have found it so, and some of my friends also.

V. THE WATER SUPPLY OF THE CITY OF NEW YORK.

By E. Waller, Ph. D.

I desire, in the first place, to present the results of complete analyses of the Croton water made at different times. The various denominations of salts quoted have been given in order to literally quote the different analysts. For the three first, double columns are given, representing the results in grains per English (Imperial) gallon of 70,000 grains, and also in grains per United States gallon of 58,318 grains, the first columns in each case being the form in which the analysts have recorded their results, to judge from the context. In Nos. 4 and 5, the magnesium and calcium bicarbonate have been calculated back to mono-carbonates, and the results given in brackets. Another table of the same results, calculated to parts per 100,000, is appended.

There is probably less difference in the results than the figures would seem to indicate, the mode of stating the results in the earlier analyses rather suggesting different methods of conducting the examination, and calculating the results to those at present in use.

Next, permit me to call your attention to a chart showing graphically the variations found in the constitution of the Croton water by Dr. C. F. Chandler during the Summer months of 1867 and 1868, representing some fifty examinations, and my own results, made in a similar manner every week, from the latter part of 1872 to the middle of 1879, representing about 350 examinations. The average results may be thus stated :

Average of results of tests made on Croton water (Parts per 100,000) :

		Mineral matter.	Org. and volatile.	Total solids.	Hardness.	Ox'n absorbed from Permanganate.
Summer of	1867	6.72	1.12	7.84	4.32	0.181
"	1868	5.66	1.97	7.63		0.168
Last 2 mos.	1872	7.48	0.44	7.92	3.553	0.131
Year	1873	6.23	1.59	7.82	3.395	0.135
"	1874	5.83	1.76	7.59	3.332	0.166
"	1875	5.656	1.835	7.491	3.293	0.211
"	1876	5.416	1.682	7.098	3.159	0.185
"	1877	5.603	1.823	7.426	3.260	0.253
"	1878	5.299	1.904	7.203	2.846	0.183
First 5 mos.	1879	5.424	0.912	6.336	2.811	0.072
Average from Nov. 1872, to May, 1879, inclusive		5.702	1.678	7.380	3.210	0.180

The "Total solids" were determined by weighing the residue obtained by evaporating a measured quantity ; the "Organic and Volatile" by igniting this residue, moistening with carbonic acid water, drying and weighing again ; "Hardness" by standard soap solution as usual, the results being expressed in the equivalent of calcium carbonate, while the "Permanganate" test was that of Miller. (J., Lond. Chem. Soc., 1865, p. 117 ; see also R. Angus Smith, Watts' Dictionary, v. 1029 ; Wauklyn's Water Anal., 1st Ed., 1868, p. 42. &c. and others), acidifying with $H_2 S O_4$, adding

standardized solution of permanganate, until the color held for half an hour at the ordinary temperature of the laboratory.

In addition to these, several other examinations made at irregular intervals, and at other times than those specified above might be given, but as they present no marked deviations, I will not occupy your time with them.

As a sample of similar determinations made on samples of the Croton, taken from different parts of the city at the same time, I would present the results obtained in April of last year, when the odors in the water caused suspicion that it contained some compounds dangerous to health.

No.	Locality.	Mineral matter.	Organic & Vol.	Total Solids.	Oxygen absorbed from Permanganate.
1	West 33d St.	6.6	2.2	8.8	0.064
2	East 34th St.	6.0	1.5	7.5	0.060 F
5	West 131st St.	4.4	1.3	5.7	0.064 F
8	East 122d St.	5.7	trace	5.7	0.062

The samples marked F were clarified by subsidence or filtration before examination, as they contained varying amounts of muddy sediment, and were therefore not fair samples of the water as ordinarily used. It may be mentioned that about a pint of No. 5 on standing half an hour in a cylinder, some $2\frac{1}{2}$ inches in diameter, deposited a sediment $\frac{3}{8}$ of an inch in depth. When this sediment was distributed as evenly as possible through the water and a portion was examined, the results were

No.	Mineral.	Org. and Vol.	Total.	Oxygen from Permanganate.
2	75.7	21	96.70	0.366
5	69.2	15.1	84.30	0.455

A portion of the sediment was examined separately. Shaken with ether it afforded as soluble in that menstruum, a minute proportion of vegetable wax having a slight greenish-brown tinge, probably from the presence of chlorophyll.

A small amount was obtained for analysis. The results were

Loss on ignition.	23.31		
Silica	43.61 to 51.00%		
Lime	0.63	$=CaCo_3$	1.12%
Magnesia	3.16	$=MgCo_3$	6.64

Ferric and Aluminic oxides 20.92

As to other determinations on the Croton Water, the following results are offered.

Parts per 100,000.

Date.	Free Ammonia.	Albummoid Ammonia.	Remarks.
August, 1874.	0.00095	0.0145	average of 6
December, 1877.	0.0010	0.0102	" of 2
Nov. 16th, 1878.	0.0015	0.0130	
July 11th, 1879.	0.0008	0.008	
April 4th, 1881.	0.0020	0.0110	
" 22d, "	0.0016	0.0117	average of 10
" " "		0.019 to 0.031	Total NH_3 on very turbid samples.
May 16th, 1881.	0.001	0.007	
Nov. 8th, 1881.	0.002	0 012	

	Nitrogen in Nitrates.
July 29th, 1881.	0.0198
Nov. 8th, "	0.0181

The results obtained on free and albuminoid ammonia do not indicate any material alteration in the proportions of those constituents yielding nitrogen in those forms since the examinations began to be made. The investigation of last April shows that the quality of the water does not vary very much in different parts of the city at the same time, unless the sediment is mixed in, when the amount of nitrogen obtainable as ammonia by distillation may reach nearly thrice the amount obtainable from the sample when fairly clear.

The amounts of nitrogen in nitrates so far as they go give no indications of sewage contamination in the water.

About the end of last year a paper by Prof. Leeds on "Relative purity of city waters in the United States," was published in the *Chemical News* (XLIV p. 265), in which the Croton water was condemned as contaminated.

The analytical results were given as follows:—

Croton, June 23d, 1881.		(Results in parts per 100,000)	
Free Ammonia	0.0027	Chlorine	0.350
Albuminoid "	0.027	Hardness	3.30
Oxygen required	0.81	Total Solids	11.80
Nitrites	none	Mineral Matter	5.00
Nitrates	0.8325	Organic & Vol.	6.80

These results I strenuously object to as misleading.

The term "nitrates" is indefinite, and when so many chemists calculate their results to "nitrogen in nitrates," &c. a false impression is readily created by the above statement in that regard. The "oxygen required," I have learned was obtained by Kubel's method —by the action of potassium permanganate on the water strongly acidified with sulphuric acid at the boiling temperature. Inasmuch as most chemists (at least in English speaking countries), use the permanganate test at ordinary temperatures,such a statement as the above, without specifying the method used, is calculated to convey a false impression of the quality of the water. Moreover the test performed in that way is open to serious objections.

Under those circumstances the chlorine in the water would affect the results, and Prof. Leeds himself has shown us that the reagents used invariably contain impurities which would affect the test to the prejudice of the water tested, the permanganate containing chlorine compounds, and the sulphuric acid (which is used in considerable amount) containing lower oxides of nitrogen and sulphur, so that where a line is drawn on comparatively small amounts of oxygen absorbed from permanganate, the impurities in the reagents would make a great difference in the conclusions to be drawn.

Prof. Leeds' results on Total Solids and Free and Albuminoid ammonia are very high, indeed higher than any results I have obtained during the past fourteen years, except when—as last spring—the samples of water were so charged with sediment as to render them by no means fair samples of the Croton Water as ordinarily obtainable. The conclusion would seem to be that his sample was turbid with sediment.

With regard to other determinations they either agree with the preceding examinations, or the methods employed were different from those of which I made use, and therefore preclude a comparison between them.

In commenting on the results, Prof. Leeds remarks: "New York and all the places mentioned lower on the list receive their water from contaminated sources. The feeders which empty into Croton Lake, the principal reservoir of the New York water, pass through a settled country, with numerous tanneries, factories, &c., along their banks. Analyses of the Croton water, made at different times during the past five years, have shown that it is to be classed among contaminated water supplies." A quotation of this statement was sent to Mr. Isaac Newton, Chief Engineer of the Croton Aqueduct Department. His reply was briefly to the effect that he

had comparatively recently examined the Croton water-shed, and that Prof. Leeds' assertion with regard to it was altogether erroneous.

From other sources, I have been able to ascertain, *first*, that with regard to the population of the water-shed: for its area of 339 square miles the population is from 17 to 20,000, or about one man to every ten acres. Permit me to quote the table given by Mr. D. M. Greene in the 23d annual report of Water Commissioners of Troy for 1877, p. 120.

Population of water sheds for city supplies :

City.	Population per square mile.
Rochester, N. Y.	36
New York, N. Y.	65
Albany, N. Y.	77
Poughkeepsie, N. Y.	86
Schnectady, Cohoes, and West Troy, N. Y. } Supply from Mohawk River.	103
Brooklyn, N. Y.	119
Boston, Mass.,	229
London, England,	270

Second.—As to industries in the Croton water-shed. But few tanneries now exist in that region for the simple reason that the most of the trees yielding the necessary bark have been cut down, and tanning is no longer profitable in that section. As regards other industries the region contains but few factories of any kind, and those are on a small scale.

To sum up, I desire to express a most emphatic dissent from Prof. Leeds' conclusions for the following reasons:

1. The proportion of chlorides existing in the water has not increased of late years so far as the records extend, and hence no indications of contamination by sewage or manufacturers can be asserted to exist.

2. The amounts of oxygen absorbed by permanguate test for a number of years, (serving to compare the water with itself at different times) show no changes in the quality of the Croton water. The same may be said for the results on Free and Alluminoid Ammonia and Organic and Volatile matter.

3. The Croton water-shed is not crowded either with population or with manufacturers as Prof Leeds seems to imagine.

4. The health of the community is not, and has never been, such as to indicate the presence of any contamination in the water supply.

COMPLETE ANALYSES OF CROTON WATER.

RESULTS IN GRAINS PER GALLON.

Number	1	1	2	2	3	3	4	5	6	7
Analyst	Professor J. C. Booth.		Dr. J. R. Chilton.				C. F. Chandler.		E. Waller.	
Date	1843.		1843.		August, 1859.		Sum'er 1869.	May, 1872.	May, 1879.	Nov. 1881.
Gallon used	Eng.	U. S.	Eng.	U. S.	Eng.	U. S.*				
Sodium chloride	....	....	0.44	0.367	4.404	0.336	0.402	0.284	0.205	0.205
Calcium sulphate	....	....	.....	.....	0.353	0.294	0.158	0.024	0.723	.....
Alkaline chlorides	0.193	0.161	.....	.....	.....	.....	.....	.....	.....	.....
Potassium sulphate	....	.....	.....	.....	.....	.....	0.179	0.205	0.188	0.201
Sodium sulphate	....	.....	.....	.....	.....	.....	0.260	0.024	0.200	0.216
Akaline carbonates	0.828	0.690	0.90	.....	0.270	0.225	.....	.....	.....	0.054
Magnesium chloride	.....	....	0.84	0.750	0.147	0.122	.....	.....	.....	.....
Calcium chloride	.....	....			0.104	0.087	.....	.....	.....	.....
Magnesium carbonate	0.939	0.782	1.52	0.700	0.390	0.325	(1.101)	(0.770)	0.918	0.685
Calcium carbonate	2.293	1.910	.....	1.266	0.836	0.696	(1.648)	(1.439)	1.650	1.319
Magnesium bicarbonate	.. .	.....	.....	... ..	... .	.....	1.913	1 338	.....	.....
Calcium bicarbonate	.....	.....	.....	.....	.....	.....	2.670	2.331	.....	.....
Ferric & aluminic oxides	0.110	0.092	.....	.....	.....	.....	trace	0.058	0.175	0.045
Silica	0.359	0.299	0.46	0.383	0.170	0.142	0.621	0.222	0.274	0.210
Organic and volatile	0.276	0.240	.....	.....	0.916	0.763	0 670	0.874	0.560	0.233
Total solids	.....	.....	.....	.....	3.590	2.990	6.873	5,360	.....	... ..
Solids by evaporation	4.998	4.174	4.16	3.466	3.705	3.087	4.780	3.849	4.893	3.168
Chlorine	.....	... ..	.....	... ..	.....	0.296	0.243	0.172	0.124	0.124

ABOVE RESULTS GIVEN IN PARTS PER HUNDRED THOUSAND.

Number	1	2	3	4	5	6	7
Analyst	Booth.	Chilton.		Chandler.		Waller.	
Date	1843.	1843.	1859.	1869.	1872.	1879.	1881.
Sodium Chloride	.....	0.629	0.577	0.690	0 487	0.351	0.351
Calcium sulphate	.....	.....	0.504	0.272	0.041	1.239	.....
Alkaline chlorides	0.276	.....	.....	.....	.....	.....	.....
Potassium sulphate	.....	.....	....	0.309	0.351	0 322	0 345
Sodium sulphate	.....	....	.....	0.449	0.041	0.343	0.371
Alkaline carbonates	1.183	... ..	0.386	... .	...	... ..	0.092
Magnesium chloride	.....	1.286	0.210	.....	.....	.....	.....
Calcium chloride	.....	.....	0.149	.....	.....	.....	.....
Magnesium carbonate	1.341	1.200	0.557	(1.888)	(1.320)	1.575	1.174
Calcium carbonate	3.276	2.171	0.194	(2.826)	(2.467)	2.830	2.262
Magnesium bicarbonate	.....	.....	.....	3.280	2.294	.....	.....
Calcium bicarbonate	.....	.....	.....	4.578	3.996	.....	.....
Ferric and aluminic oxides	0.157	... .	0.243	trace.	0.100	0.300	0 078
Silica	0 513	0.657		1.060	0.380	0.470	3.360
Organic and volatile	0.394	....	...	1.060	0.380	0.470	3.360
	.....	... .	1.309	1.150	1.500	0.930	0.400
Total	.....	.....	5.129	11.788	9.190	.....	.....
Solids by evaporation	7.140	5.943	5.293	8.200	6.600	8.390	5.432
Chlorine	....	....	0.508	0.416	0.294	0.213	0.213

Nos. 1 and 2. Illustrations of the Croton Aqueduct. F. B. Tower. N. Y., 1843, p. 135,
No. 3. Report of Water Commissioners of Albany for 1865, p. 50.
Nos. 4 and 5. Report of Board of Health for 1871, New York, p. 371.
No. 6. Report on Croton Water, New York, 1881, p. 45.
* U. S. gallon of 231 cubic inches (58,318 grains).

VI. HEPTYLENE FROM HEPTANE OF P. SABINIANA.

By F. P. Venable, Ph. D.

University of North Carolina.

In a note on certain heptylic derivatives published in 1880*, mention was made of a heptylene agreeing in boiling point with the one prepared by Schorlemmer from the petroleum-heptane. This heptylene was observed as one of the products in preparing several of the heptylic derivatives from the bromide, but the method adopted for its special preparation was the action of sodium ethylate upon the heptylic bromide at the temperature of boiling. The liquid obtained was light, mobile and with a high refractive-index, boiling at 97°–98°C, and possessing a pleasant aromatic smell. Its specific gravity was 0.70075 (21°. 6 C).

If sodium ethylate is allowed to act in the cold, a heptylethyl-ether is formed, together with a small amount of heptylene. Even when an excess of the sodium ethylate is present, it is difficult to change all of the bromide to the ether. Analyses, however, of the heavy oily liquid obtained showed the conversion to be almost complete

HEPTYLENE BROMIDE.

The action of bromine upon the heptane is slow, and heat must be applied. On the heptylene, bromine acts with great energy, heat being evolved. In the first case, the resulting product is one of substitution; in the latter, it is one of addition; but substitution products are very easily formed if the temperature is allowed to rise. In the brominating, then, it is necessary to avoid all rise of temperature. The heptylene was put in a long tube surrounded by a freezing mixture of sodium sulphate and hydrochloric acid, and air loaded with bromine vapor was sucked through it for six or ten hours. The color of the bromine disappeared with each bubble, and the reaction was stopped as soonas a permanent coloration was observed. The thick, oily liquid was then distilled in partial vacuum, boiling quite constantly without the evolution of hydrobromic acid gas. After the first distillation it was found possible to complete the fractioning under ordinary pressure. The boiling point was 209°–211° C. (uncorrected.)

Only with great difficulty did Schorlemmer † succeed in

* Ber. Chem. Ges. XIII. 1649.

† Annalen Chem. Pharm. 188, 256.

forming a heptylene bromide or heptylic dibromide from his petroleum-heptane, and it could not be distilled. The dibromide prepared by Thorpe and Young ‡ has a spec. grav. 1.5146 (18.5° C), and underwent decomposition at the temperature of 150° C—that is, considerably below the boiling point as observed for the dibromide described above.

I propose carrying the investigation of this heptylene and its derivatives still further, as their stability gives promise of interesting results. A careful determination of the specific gravity of the the dibromide will also be made as soon as a larger supply is on hand.

‡ Annalen Chem. Pharm. 165, 12.

PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

Room 1, *University Building, N. Y., March* 3*d*, 1882.

The meeting was called to order at 8:20 P. M. Prof. A. R. Leeds in the chair.

The minutes of the previous meeting were read and approved.

No report was received from the Board of Directors.

Dr. E. Hirshberg was then elected a regular member.

After which the following gentlemen were nominated as members of the Society:

Thos. S. Gladding as regular member. Proposed by Chas. M. Stillwell, T. J. Parker and A. R. Leeds.

Dr. G. Krause as regular member. Proposed by Geo. A. Prochazka, A R. Leeds, and James H. Stebbins, Jr.

Robert G. Remsen, Jr., M. D., as regular member. Proposed by A. R. Leeds, James H. Stebbins, Jr., and A. H. Elliott.

Prof. A. A. Brennerman as regular member. Proposed by A. R. Leeds, James H. Stebbins, Jr., and A. H. Elliott.

G. E. Perkins as regular member. Proposed by T. O'C. Sloane, M. Benjamin and W. Rupp.

After which Mr. Elliott moved that the regular order of business be suspended, in order to give more time to discuss the publishing of the Journal.

Carried.

The Secretary then moved that the above discussion be postponed to 9 P. M., in order that some of the papers of the evening might be read.

Carried.

The first paper of the evening: "On the Determination of Zinc as Pyrophosphate," by Mr. Geo. C. Stone, was then read.

After some remarks by the Chairman, Dr. Sloane read his first paper "On Methyl Violet Test Paper."

Some remarks by Dr. Grothe followed.

After which Dr. Sloane read his second paper: "On a Fat Extraction Apparatus."

The third paper: "Upon the Separation of Alumina and Sesquioxide of Iron from Manganese," by Mr. Nelson H. Darton, then followed.

Dr. Endemann stated that Drs. Behr and Ledoux had both

resigned from the Committee on Papers and Publications, and that he now, officially, tendered his own resignation as Editor; but stated that he would remain Editor till the next number of the Journal is published.

He furthermore stated that he did not think it would be wise to increase the Committee on Papers and Publications to five, as he thought three competent men would be sufficient.

After a good many remarks, both for and against the proposed change, it was finally moved that this subject be referred to the Board of Directors, who in turn shall recommend their decision to the Society.

Carried.

After which it was moved that the reading of the papers should be proceeded with.

Carried.

Dr. Waller then took the chair.

The fourth paper: "On Some New Compounds of Diphenylamine," by Prof. A. R. Leeds, was then read.

After which the fifth paper: "On Methods of Water Analysis and the Character of the New York Water Supply," by Prof. A. R. Leeds was read.

Dr. Waller remarked that he also considered the permanganate method of Mille as unsatisfactory when applied to isolated samples, but that he considered it of value when the same water is compared with itself at different times. He also objected to Kubel's method, on the grounds previously mentioned in his paper. He did not mean to criticize unfavorably Dr. Leeds method for "mineral matter," but preferred to use carbonic acid water after ignition.

As the hour was late it was moved that the paper of Mr. Gladding on Reverted Phosphates be made a special order of business at the next Conversazione.

Carried.

After which the meeting adjourned.

JAMES H. STEBBINS, JR.,

Recording Secretary.

VII. DETERMINATION OF ZINC AS PYROPHOSPHATE.

By Geo. C. Stone, Ph. B.

Sometime since I had occasion to analyze a sample of water said to contain the tailings from an amalgamating mill. The analysis of the suspended matter only added up about eighty-eight per cent. I tried boiling the filtrate from the magnesia with KOH, this threw down a fine, crystalline, white precipitate; on making a qualitative examination of this I found it contained ZnO, and P_2O_5. On weighing the precipitate and calculating the amount of ZnO. it contained, supposing it to be $Zn_2 P_2 O_7$, I found it eleven and a fraction per cent., which made my analysis add up very close to one hundred per cent. This led me to experiment with phosphate of zinc with the following results.

If a mixture of an acid solution of a zinc salt and an alkaline ortho-phosphate is exactly neutralized with ammonia, the zinc is precipitated as a bulky, flocculent double phosphate of zinc and ammonia, easily soluble in acids or ammonia, and slightly soluble in potassic hydrate. If the solution in ammonia is boiled, thedouble. phosphate reprecipitates as a fine, crystalline white precipitate, very slightly soluble in ammonia, and almost absolutely insoluble in water. If the boiling is continued till the free ammonia is all driven off, the precipitation is complete. On drying and igniting this precipitate the zinc is obtained as pyrophosphate $Zn_2 P_2 O_7$.

This method of determining zinc has several advantages over the one commonly employed, of precipitating as sulphide, dissolving in hydrochloric acid, reprecipitating as basic carbonate, igniting and weighing as oxide.

First.—It is much quicker, as it is not necessary to let the precipitation stand, and there is only one precipitation.

Second.—The precipitate settles rapidly, filters quickly, does not run through the filter, washes easily, dries quickly, brushes off the paper easily and completely when dry, and is very difficult to reduce.

Third.—The presence of ammonia salts does not interfere with the completeness of the precipitation.

In all of which particulars its behavior is quite the reverse of that of either the sulphide or basic carbonate.

Fourth.—The pyrophosphate contains only 42.76 per cent. of zinc, while the oxide contains 80.25 per cent. so that an error in weighing makes much less difference in the result.

Fifth.—The phosphate does not need any purification, whereas the oxide frequently contains silica.

On the other hand the phosphate bumps badly while boiling out the ammonia; and the precipitate sticks pretty tightly to the beaker. If the solution is kept rather dilute and only just enough ammonia is added to redissolve the precipitate first formed, and the solution is frequently stirred, the bumping will not give much trouble. The precipitate that sticks to the beaker is best removed by dissolving in a few drops of HCl, adding a little $NaNH_4HPO_4$, making just alkaline with NH_4OH and boiling till the free ammonia is expelled.

Experiment I.—Was made more to test the behavior of the phosphate of zinc and to see if the results agreed, than to test the absolute accuracy of the method. 0. 2 grms. of zinc, coated with oxide, were dissolved in H_2SO_4 and a few drops of HNO_3, the solution was diluted to about 300 cc., an excess of $NaNH_4HPO_4$ added, and just enough NH_4OH to re-dissolve the precipitate first formed. The solution was then boiled till there was no longer any smell of NH_3, filtered, washed thoroughly with hot water, dried, ignited and weighed. Gave:

A $Zn_2P_2O_7$ 0.4627 grms.=Zn 0.1979 grms.
B $Zn_2P_2O_7$ 0.4624 grms.=Zn 0. 1977 grms.

A was filtered as soon as the NH_3 was expelled. B was allowed to stand in a warm place for three hours after boiling.

Experiment II.—0.2 grms. of zinc ore, mainly oxide, was dissolved in HCl, HNO_3 and H_2SO_4, evaporated till the HCl and HNO_3 were expelled, diluted, filtered, the iron separated as basic acetate, dissolved and reprecipitated by a large excess of NH_4OH, the filtrates combined and evaporated to about 300 cc., an excess of $NaNH_4HPO_4$ added and the zinc determined as in I. In a second portion of 1. 0 grm. the iron was separated as above, the zinc precipitated by H_2S in a boiling acetic acid solution, let stand, filtered, washed, dissolved in HCl, reprecipitated as basic carbonate by Na_2CO_3, ignited and weighed as oxide. Gave:

A $Zn_2P_2O_7$ 0.3441 grms. Zn 0.1471 grms.=73.55 per cent
B $Zn_2P_2O_7$ 0. 9149 grms. Zn 0.7341 grms.=73.41 " "

Experiment III.—0.2 Grms. ferruginous blende treated as in II and gave:

A $Zn_2P_2O_7$ 0.2721 grms.=Zn 58.17 per cent
B $Zn_2P_2O_7$ 0.2716 grms.=Zn 58.07 " "

The analysis was afterwards completed and gave :

Zn	(average of 2 determinations)	58.12 per cent
Fe	" " "	7.61 " "
Insoluble silicious matter		1.43 " "
S calculated to give Fe S and Zn S		32.96 " "
Total,		100.12

Experiment IV.—1.0 grm. Zn $(C_2H_3O_2)_2$ 2 H_2O dissolved in water, excess of $NaNH_4HPO_4$ and NH_4OH added, the NH_3 expelled by boiling and the zinc determined as before. Gave :

A $Zn_2P_2O_7$ 0.6953 grms.=Zn 29.78 per cent
B $Zn_2P_2O_7$ 0.6974 grms.=Zn 29.82 " "

The theoretical percentage is 29.68.

Experiment V.—To test the influence of alkaline and ammonia salts :

0.5 grms. Zn $(C_2H_3O_2)_2 2H_2O$, 2.0 grms. Na_2CO_3, 2.0 grms. NH_4Cl, and 5.0 grms. Na $C_2H_3O_2$
the zinc determined as before gave :

A $Zn_2P_2O_7$ 0.3484 grms. = Zn 29.78 per cent.
B $Zn_2P_2O_7$ 0.3469 grms. = Zn 29.66 per cent.

Experiment VI.—As the phosphate of zinc is soluble in NH_4OH, while the phosphates of lime, magnesia, and manganese are insoluble in it, it seemed probable that the zinc could be separated by means of this reaction. The following experiments were tried to test this : 0.5 grm. Zn $(C_2H_3O_2)_2$ 2 H_2O, 0.1 grm. Mn_3O_4, 0.1 grm. $CaCO_3$ and 0.1 grm. $MgSO_4$ $7H_2O$ were dissolved in HCl excess of Na NH_4HPO_4 and NH_4OH added and let stand cold for eighteen hours, the clear solution filtered off, the precipitate dissolved in HCl, reprecipitated by NH_4OH and a few drops of Na NH_4HPO_4, filtered, washed with cold dilute NH_4OH, the NH_3 boiled out, and the zinc determined as before, gave:

A $Zn_2P_2O_7$ 0.3444 grms. = Zn 29.46 per cent.
B $Zn_2P_2O_7$ 0.3451 grms. = Zn 29.52 per cent.

The phosphates of manganese and lime clogged the filter and showed a tendency to run through it. To get good results the solution must be rather dilute (400 to 500 cc.).

Experiment VII.—0.5 grms. Zn $(C_2H_3O_2)_2$ $2H_2O$ and 0.5 grms. $MgSO_4$ $7H_2O$, treated as in the last experiment, gave:

A $Zn_2P_2O_7$ 0.3453 = Zn 29.54 per cent.
B $Zn_2P_2O_7$ 0.3451 = Zn 29.52 per cent.

Experiment VIII.—As phosphate of lime filters very slowly and is very difficult to wash, I tried precipitating the lime as oxalate in a rather dilute solution, decanting, dissolving in HCl, reprecipitating, and determining the zinc in the filtrate as phosphate. 0.5 grms. Zn $(C_2H_3O_2)_1$ $2H_2O$ and 0.25 grms. $CaCO_3$ taken, gave:

$Zu_2P_2O_7$ 0.3429 grns. = 29.32 per cent.

Experiment IX.—1,365 grms. $Zn_2P_2O_7$, heated to redness for about half an hour, weighed 1.365 grms.

Experiment X.—0.4 grms. zinc ore, containing about 14 per cent. SiO_2, 20 per cent. Fe_2O_3, 12 per cent. $CaCO_3$, and 16 per cent. MnO. A. Iron separated as basic acetate, dissolved and reprecipitated. Manganese precipitated by Br. Excess of $NaNH_4HPO_4$ and NH_4OH added, the solution decanted from the lime, precipitate, dissolved and reprecipitated by a large excess of NH_4OH, filtered and washed with dilute NH_4OH, using a filter pump, the excess of NH_3 boiled out, and the zinc ignited and weighed as $Zn_2P_2O_7$.

A. $Zn_2P_2O_7$ 0.2760, Zn 0.1179 = 29.47 per cent.

B. Iron precipitated as basic acetate, dissolved and reprecipitated by a large excess of NH_4OH, the filtrates combined, made strongly acid with $HC_2H_3O_2$, the zinc precipitated by H_2S in a boiling solution, filtered, dissolved in HCl and $KClO_3$, reprecipitated by Na_2CO_3, ignited, and weighed as ZnO, gave:

B. ZnO 0.1466, Zn 0.1176 = 29.40 per cent.

Experiment XI.—0.4 grms. ore similar to the last, treated in the same way, gave:

A. $Zn_2P_2O_7$ 0.2468 Zn 0.1055 = 26.37 per cent.
B. ZnO 0.1303 Zn 0.1045 = 26.15 per cent.

Experiment XII.—0.4 grms. ore similar to the last, treated in the same manner, gave:

A. $Zn_2P_2O_7$ 0.2390, Zn 0.1022 = 25.55 per cent.
B. ZnO 0.1294, Zn 0.1030 = 25.75 per cent.

The results of the separations are not as good as I could wish; but I intend to continue the experiments, in hopes of improving on them.

RESUME:

	Substance Analyzed.	Grms. used.	Grms Zn found.	REMARKS.
Expt. I.	Impure Zinc.	0.2	0.1979	
"	" "	0.2	0.1977	
" II.	Zinc Ore.	0.2	0.1471	Weighed as $Zn_2P_2O_7$
"	" "	1.0	0.7341	" " ZnO
" III.	Blende	0.2	0.11634	
"	"	0.2	0.11614	
" IV.	$Zn(C_2H_3O_2)_2$ $2H_2O$	0.1	0.2978	Theory requires 0.2968 grms Zn.
" V.	"	0.1	0.2982	+2 grms. NH_4Cl+2 grms. Na_2CO_3+5 grms. $NaC_2H_3O_2$
"	"	0.5	0.1489	
" VI.	"	0.5	0.1484	+0.1 grm. Mn_3O_4+0.1 grm. $CaCO_3$+0 1 grm. $MgSO_4, 7H_2O$
"	"	0.5	0.1473	
" VII.	"	0.5	0.1476	+0.5 grms. $MgSO_4, 7H_2O$
"	"	0.5	0.1477	
" VIII.	"	0.5	0.1476	+0. 25 grms. $CaCO_3$ Separated as oxalate.
"	"	0.5	0.1466	
" IX.	$Zn_2P_2O_7$	1.365	1.365	Ignited for half an hour.
" X.	Zinc Ore.	0.4	0.1179	Weighed as $Zn_2P_2O_7$
	" "	0.4	0.1176	" " ZnO
" XI.	" "	0.4	0.1055	" " $Zn_2P_2O_7$
	" "	0.4	0.1046	" " ZnO
" XII.	" "	0.4	0.1022	" " $Zn_2P_2O_7$
	" "	0.4	0.1030	" " ZnO

Since making the above experiments, I have found that Hugo Tamm(*Chem, News* XXIV, p. 148) has proposed to weigh zinc as the double phosphate of zinc and ammonia, $Zn\,NH_4\,PO_4$, which, of course, necessitates the use of a weighed filter. His method is as follows: Make acid solution of a zinc salt alkaline with $N\,H_4O\,H$, then barely acid with HCl and add a solution of $Na_2\,HPO_4$ heat nearly to boiling for a few minutes, filter, wash, dry at 100°C. and weigh as $Zn\,NH_4\,P\,O_4$.

He states that there is invariably a loss when the double phosphate is converted into pyrophosphate by ignition ; but from the results of my own experiments, I should say the loss was due to his precipitate being formed in an acid solution, in which I have found the double phosphate to be quite soluble. The greater quantity of zinc found when weighing the double phosphate was probably due to insufficient heat in the drying. He uses the atomic weight of 65.5 for zinc ; using 65, the one now generally accepted, his results would all be low.

The composition of pyrophosphate of zinc is as follows:

2 Zn	130	42.763 per cent.
2 P	62	20.395 "
7 O	112	36.842 "
$Zn_2P_2O_7$	304	100.000

or,

2 ZnO	162	53.29 per cent.
P_2O_5	142	46.71 "
$Zn_2P_2O_7$	304	100.00 "

Nickel and cobalt form phosphates which behave precisely like the corresponding zinc salt; both, however, are strongly colored, and so can readily be distinguished from phosphate of zinc.

VIII. METHYL VIOLET TEST PAPER.

By T. O'Connor Sloane A. M., Ph. D.

Several years have passed since it was first proposed to use methyl violet as an indicator or test for mineral acids in the presence of organic acids. Since it has also been used as a coloring matter for test paper it occurred to me that a few notes on the preparation and use of the paper would be of interest.

I tried solutions of methyl violet of different strength in dyeing paper. A solution of one part of violet in four thousand parts of water was found to be sufficiently strong. A solution of double this strength gave a very good color.

Using paper dyed with the first solution I performed the following experiments:

One cc. of concentrated hydrochloric acid was mixed with him one hundred cc. of water. The acid was of sp. gr. 1.197 corresponding to about 40 per cent. acid; the solution therefore was of four one thousands strength or four-tenths of one per cent.

A piece of paper dipped in this solution did not show the reaction except on drying; then it came out perfectly. A piece made from a violet solution of half the strength showed a little; this acid seemed therefore to be close upon the limits of sensibility for an immediate reaction.

It will also be noticed that the more weakly colored paper was slightly more sensitive; on drying the darker paper shows as well or better.

The acid was diluted to one-fifth its former strength or eight hundreths of one per cent. I now adopted the following way of operating: A convex cover was plaeed on a water bath, the water in which was heated to boiling; each piece of paper as dipped was laid on the upper convex surface of the cover; this dried it in about one minute; in practice this will be found an excellent way of using the paper.

The standard paper dipped once and dried showed the reaction, but not strongly; a second dipping and drying brought it out perfectly; a third treatment made the reaction still stronger.

When a paper colored by this weak acid was dipped a second time the previous change of color was indiscernible until it had dried again.

The acid was reduced to four-hundredths of one per cent.; the paper just showed the reaction after one dipping and drying; a second treatment developed it perfectly.

The acid was next reduced to two hundredths of one per cent. strength; the reaction appeared after three to five treatments but very faintly.

About the same results were obtained with sulphuric acid of corresponding strengths.

The series of experiments indicates pretty closely the limits of sensitiveness; drying the paper cannot be too strongly insisted upon; independent of any concentration of acid thus effected, the color change which is masked to a great extent by moisture is made visible by drying.

IX. DIPHENYLAMINE—ACROLEIN.

By Albert R. Leeds.

25 grms. of diphenylamine, in alcoholic solution, were treated with acrolein in excess, and after standing, the loosely-corked flask was gently warmed for a number of hours, until the smell of acrolein had nearly disappeared. A bulky dark red precipitete was formed. On boiling with alcohol a deep red solution was obtained and the portion undissolved formed a tenacious sticky mass, very awkward to work with. By repeated boiling with water under a return cooler this mass gradually lost its sticky nature. It was then digested alternately with boiling water and alcohol, until at last the mass became pulverulent and could be ground up in a mortar. The operation of boiling was then repeated many times, the

mass being powdered after each treatment with water, until at length the substance in a state of purity was obtained. Its analysis showed it to be Diphenylamine–Acrolein or, as it might be termed Didiphenylamineallyline, $(C_{12}H_{10}N_2)\ C_3H_4$.

	Found	Theory
Carbon,	86.26	86.18
Hydrogen,	6.29	6.36
Nitrogen,	7.28	7.45

It does not melt or sublime but is decomposed on breaking, leaving behind a carbonaceous residue extremely difficult of combustion. It is very slightly soluble in alcohol, insoluble in ether, and readily soluble in chloroform to a dark red liquid. From this solution, and also from that in glacial acid, in which it dissolves to a red liquid, but less readily than in chloroform, it could not be made to crystallize.

Its solution in chloroform was attacked with great energy by bromine. So also its solution in acetic acid, a dark red compound being formed on the addition of two atoms of bromide to one molecule of the diphenylamine. This compound was readily soluble in chloroform, but as it did not separate in a crystalline condition its anlaysis was not made. It was probably the addition compound $(C_{12}H_{10}N_2)C_3H_4Br_2$.

Its solution in acetic acid was attacked by nitric acid, forming a precipitate with a supernatant yellow liquid. Neither the solution or the precipitate yielded a crystallizable nitro-product, and their study was abandoned.

PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

April 7th, 1882.

Room 1, *University Building.*

The meeting was called to order at 8.30 P. M.

Prof. Chas. A. Doremus in the chair.

The minutes of the previous meeting were read and approved.

The minutes of the Board of Directors were then read and accepted.

Mr. Elliott then moved that the regular order of business be suspended, in order to give more time for the election of the new committee on Papers and Publications.

Carried.

The Recording Secretary then stated that Messrs. Waller, Doremus and Friedburg had consented to serve on said committee, and he presented their names to be acted on.

After balloting, the above gentlemen were declared elected.

The recommendation of the Board of Directors that the Committee on Papers and Publications be authorized to issue a monthly number of the journal containing as much matter as the funds on hand will warrant, was carried unanimously.

After which the following gentlemen were elected as regular members of the Society.

Dr. G. Krause—Proposed by Geo. A. Prochazka, A. R. Leeds and James H. Stebbins, Jr.

Thos. S. Gladding—Proposed by Chas. M. Stillwell, T. J. Parker, and A. R. Leeds.

Prof. A. A. Brenneman—Proposed by A. R. Leeds, James H. Stebbins, Jr., and A. H. Elliott.

Geo. E. Perkins—Proposed by T. O'C. Sloane, M. Benjamin and W. Rupp.

Robert G. Remsen, Jr., M. D.—Proposed by A. R. Leeds, James H. Stebbins, Jr., and A. H. Elliott.

The following nomination was made:

G. E. Bailey as regular member—Proposed by M. Benjamin, W. Rupp and A. H. Elliott after which the first paper of the evening "On a new Volumetric process for Copper," by Mr. J. Casamajor was read.

This process consisted in precipitating the copper with an alkaline sulphide solution. The precipitate conglomerates, leav-

ing a clear liquid above so that by letting the alkaline sulphide run in slowly the point may be observed where no further precipitate is formed.

After some remarks by the chair, the second paper, "Upon the determination of tannic acid," by Mr. Nelson H. Darton then followed.

Dr. Grothe then stated that he together with Dr. Friedburg had recently observed the formation of a chloroform hydrate.

Some remarks and questions by the Recording Secretary then followed.

Dr. Friedburg then moved that members should be instructed to send all their papers and manuscripts to the Committee on Papers and Publications for their approval before they are read.

Carried unanimously.

Mr. Casamajor then presented the resignation of Mr. W. Schrage which was accepted.

Mr. Casamajor stated that Prof. Mallett had accepted an invitation for our coming annual dinner and moved that a committee of three be appointed to make arrangements for said dinner.

Carried.

The Recording Secretary then moved that the Chair appoint said committee.

Carried.

The Chair, after due consideration, appointed Messrs. Casamajor, Sloane and Stebbins.

Mr. Casamajor further stated that the cost for the dinner would be $3 per plate.

After which the meeting adjourned.

JAMES H. STEBBINS, JR.,
Recording Secretary.

X. VOLUMETRIC ESTIMATION OF COPPER AND OF LEAD

By P. CASAMAJOR.

In this process, copper is precipitated from an alkaline solution by a titrated solution of sodic sulphide. This reagent is used in a process published some thirty years ago by Pelouze, in the *Annales de Chimie et de Physique.* In Pelouze's process, copper is dissolved in a large excess of ammonia, and an intensely blue liquid is obtained. To this a titrated solution of sodic sulphide is added until the blue color disappears.

To titrate the sodic sulphide, one gramme of pure copper is dissolved in nitric acid, and 40 or 50 cubic centimetres of concentrated ammonia are added, the whole being placed in a flask. This liquid is heated to boiling, and the solution of sodic sulphide is dropped in the flask until the blue color disappears. To observe this, the precipitate is allowed to settle, and more ammonia is added if necessary.

The solution of sodic sulphide was found to keep well in tightly stopped bottles.

I have had occasion to use this process several times, and have always found that the end of the reaction is not definite. The brown color of the sulphide, of which particles remain suspended, interferes with the observation of the color of the liquid. After the blue color has disappeared, an addition of alkaline sulphide will show a visible turbidity, even when copper is the only metal present.

Instead of treating a cupric solution by an excess of ammonia, I use an alkaline tartrate, dissolved in an excess of caustic soda. This liquid is the same, which, added to a titrated solution of cupric sulphate, forms Fehling's solution. It is prepared by dissolving 173 grammes of Rochelle Salt in 480 cc. of caustic soda solution, of specific gravity 1.14, and adding water sufficiently to form 1 litre of liquid. This solution is added to the acid solution of copper to be tested, in slight excess of the quantity sufficient to redissolve the cupric oxide, and to obtain a clear blue solution. The liquid in the porcelain dish is then heated to nearly boiling point, and the sodic sulphide solution is gradually added, until no turbidity is produced by the addition of one drop of the reagent.

In this manner of proceeding, the blue color of the solution is not taken into account. The brown cloud which follows the addition of the titrated reagent is the only guide. The first portion of sodic sulphide, added to the alkaline cupric solution, gives rise to an intense black brown precipitate. As soon as this is formed the liquid in the porcelain dish is thoroughly stirred with a glass rod, which has the effect of agglomerating the cupric sulphide into a coarse curdy precipitate, which rapidly settles to the bottom of the dish, leaving the liquid quite clear and nearly colorless. If, after settling, the liquid should not be sufficiently clear, it should be vigorously stirred again, until the desired effect is obtained. After every addition of sulphide of sodium, the thorough agitation of the liquid is repeated until it is sufficiently clear. The degree of turbidity caused

by the addition of the reagent is a guide as to the quantity to be added. At the beginning, the brown color is very intense, and it is useless to wait for a complete clearing up before adding more of the reagent. Towards the end, the brown cloud is very slight, and the sodic sulphide should be added slowly, stirring up after every addition. Finally the sodic sulphide should be added drop by drop, until not the faintest cloud is seen after adding one drop.

By thoroughly stirring the liquid in the porcelain dish, and thereby causing the precipitate to become agglomerated in heavy clots, the surface of the dish remains very clean. On the white porcelain, as a back ground, the faintest cloudiness is easily observed, and a liquid containing one gramme of copper in 30,000 cubic centimeters, will still give a distinct brown cloud by the addition of one drop of the reagent.

For the volumetric estimation of lead, the same process may be applied. Sulphate of lead is easily dissolved in the alkaline tartrate solution. The sulphide of lead is precipitated in exactly the same manner as sulphide of copper. The precipitate agglomerates into curd as easily as does cupric sulphide, and the end of the reaction is equally definite.

As lead can be separated from other metals as sulphate, which is easily dissolved in the alkaline tartrate solution, we have by this method a rapid way of estimating lead. The precipitation by sodic sulphide is a quick operation, which only takes a few minutes. Sulphuric acid can also be separated as plumbic sulphate and estimated in the same way. Other acids which form insoluble plumbic salts could also be estimated by this process.

Copper can be separated from every other metal as sulphocyanate (Rivot's process). This precipitate may be heated with excess of nitric acid, and the alkaline tartrate solution added, after which the copper can be precipitated as sulphide.

These volumetric processes for copper and lead are founded on the easy agglomeration into heavy clots of the sulphides of these metals by agitation, the liquid remaining perfectly clear. Other metals, whose sulphides agglomerate in the same way in the alkaline tartrate solution, can probably be estimated by this process.

An attempt was made with silver, but chloride of silver was found to be insoluble in the alkaline tartrate solution. Chloride of silver was dissolved in excess of ammonia, and sodic sulphide added, but this did not prove satisfactory as sulphide of silver is sufficiently

soluble in ammonia to give a brown solution in which a faint cloud cannot be seen.

As chloride of silver is agglomerated into heavy clots by agitation, like cupric and plumbic sulphides, the white-cloud obtained, by pouring a titrated solution of sodic chloride into a solution of nitrate of silver, may be observed against a black background. This may be had by carrying on the precipitation in a flat dish of black glass, or in one of clear white glass, the under side of which has been covered with lamp black. The solution never clears up so completely by agitation as the alkaline solutions of copper and lead, but the slight milkiness of the liquid does interfere with the observation of the white cloud due to the formation of even very small quantities of chloride of silver.

The best temperature for the agglomeration of chloride of silver is about 65° C.

FOREIGN PATENTS.

Condensed from R. Biedermann's Report to the German Chemical Society, by Otto H. Krause.

A. R. Pechiney, Salindres: *Manufacture of sodium bicarbonate* (Eng. P. 2098, May 24, 1880). This invention relates to the manufacture of soda by the ammonia process. The apparatus employed consists of a horizontal cylinder, which can be turned upon its long axis, and which is divided, first, by a perforated diaphragm parallel to the latter, and then into several compartments by discs placed at right angles to this. The discs have openings in the centre. The axles upon which the cylinder turns are hollow, to allow the gases and liquids to enter and pass out. Ammoniacal solution of sodium chloride enters, and unabsorbed gases pass out through the axle at one end, whilst the axle at the other end, which has two concentric openings, serves for the exit of the liquid containing the suspended sodium bicarbonate and for the entry of the pure carbonic acid resulting from the calcination of the latter. Impure carbonic acid, from kilns or furnaces, enters the outer concentric opening, and is led by means of pipes through the first three compartments of the cylinder and delivered into the fourth. The cylinder makes half revolutions at regular intervals. Ammoniacal solution of sodium chloride fills the apparatus, flowing through the holes in the discs, and passes into it continuously, whilst carbonic acid enters at the other end. On its way through, the liquid meets a current of gas, which latter gradually becomes richer in carbonic acid, the last three compartments receiving the pure gas.

R. I. T., N. T. and H. L. HAWKSLY, Oldham: *Forming salt into balls*, (Eng. P. 3789, Sept. 18, 1880). Salt intended for the manufacture of soda is formed into balls by means of a press, which also delivers them automatically upon the bottom of the furnace without breaking them.

W. THOMPSON, London: *Manufacture of white lead*, (Eng. P. 4056, Oct. 6, 1880.) Lead is placed in a chamber, the temperature of which is kept at 27— 50° C. Vessels containing dilute acetic acid are placed on the floor. Carbonic acid and air enter through pipes placed above to complete the reaction.

WILHELM KAYSER, Ilmena: *Material for preventing boiler incrustation*, (Germ. P. 14189, Sept. 26, 1880.) Corkwood is pulverized and impregnated with barium chloride, sodium carbonate, or other substance suited to the water to which it is to be applied. The material so prepared pervades the water evenly, and gradually deposits after receiving the precipitate upon itself.

H. KOLBE, Hamburg: *Mercurial thermometer with electric alarm and variable contact*, (Germ. P. 13166, June 4, 1880.)

T. B. LIGHTFOOT, Dartford: *Cooling apparatus*, (Eng. P. 4065, Oct. 6, 1880.) An air ice machine in which the compressed and cooled air is expanded in two separate stages. In the first stage it is only expanded to such a degree as to deposit the greater part of its moisture. In the second stage it is then further expanded.

A. R. PECHINEY, Salindres: *Calcination of sodium bicarbonate in the ammonia soda process*, (Eng. P. 2099, May 24, 1880.) The invention consists essentially in heating the bicarbonate by means of heated carbonic acid resulting from a previous operation. This is done in four chambers of masonry enclosed in an iron casing which is pivoted upon the piston of a hydraulic press so that the whole can be lifted several inches. The heated carbonic acid gas is passed into the first compartment through the bottom, and heats the sodium bicarbonate placed on a grate. Through flues in the masonry the gas then passes in the same way through the second and third compartments, whilst the fourth is being discharged and refilled. When the contents of the first chamber has been sufficiently heated the apparatus is lifted and a quarter revolution given to it. The second chamber is then put into direct communication with the heated carbonic acid, etc. The carbonic acid evolved from the apparatus is passed through condensers to eliminate the water and then into holders to be used over again.

A. R. PECHINEY, Salindres: *Separation of liquids from precipitates*, (Eng. P. 2100, May 24, 1880). Consists of a filtering cylinder for separating the liquid from precipitated sodium bicarbonate by means of hydraulic pressure.

W., T., and J. CHADWICK, Manchester, and I. W. KYNASTON, St. Helens: *Separation of iron from aluminium sulphate, &c* (Germ. P. 14185, Aug. 31, 1880; addition to G. P. 11137; see this Journal, vol. II, p. 440, and vol. III, p. 72). To separate the iron completely the inventors add calcium ferrocyanide. To facilitate the settling of the Prussian blue formed, small quantities of a metallic salt are added. Preference is given to cupric sulphate, as any traces of it which may have remained in the solution can subsequently be removed together with the arsenic with hydrogen sulphide.

F. G. SPONNAGEL, Berlin: *Improvements in the manufacture of silicate of soda*, (Germ. P. 14182, Aug. 10, 1880). Precipitation from a concentrated solution by means of ether in a hermetically closed vessel.

W. CONRAD, Wurzburg: *Method of preparing blue coloring matters from the sulphonic acids which are formed by the action of ammonium sulphite upon nitroso derivatives of tertiary aromatic monamines*, (Germ. P. 14014, Jan. 20, 1880). By allowing ammonium sulphite to react upon nitrosodimethylaniline, the sulphonic acid of the reduced base is generated. By treating the latter with hydrogen sulphide and ferric chloride (Lauth, Ber. dent. chem. Gesellsch. 1866, p. 1035) a blue coloring matter is produced.

C. TIMMERMAN, Braunschweig, and R. GRAETZEL, Hanover: *Method and apparatus for the preparation of eupittonic acid from wood tar*, (Germ. P. 13787, Nov. 12, 1880; addition to Germ. P. 9328, see this Journal, vol. II. p. 301). Differs from the original patent chiefly in the manner of oxidizing the precipitated redissolved and dried dimethyl ethers of pyrogallic and methylpyrogallic acids. The air being passed over the powder heated to 160—220° C., instead of passing it through its solution in alkali.

A. DOMEYER and J. MARZELL, London: *Preparation of pure artificial alizarine*, (Eng. P. 2784, July 7, 1880). The melt from the anthraquinone-sulphonic acid is treated with water and the alkali neutralized with sulphurous acid. After separating the precipitated alzarine, the filtrate is rendered caustic by boiling with lime and the sulphurous acid recovered as calcium sulphite.

E. JACOBSEN, BERLIN: *Method of preparing benzoic acid, benzoic ether and benzaldehyde together with organic acid chlorides, acid anhydrides, &c., from benzo tri-chloride or dichloride in presence of certain metals, metallic oxides and salts*, (Germ. P. 13128, May 25, 1880; addition to Germ. P. 11494, see this Journal, vol II. p. 462). The oxides, sulphides, hydroxides or other salts of the metals mentioned in the original patent can also be used in the processes there described.

E. PORION and L. MEHAY, Wardreques, France: *Manufacture of Alcohol*, (Eng. P. 2736, July 3, 1880). The raw material is sac-

charified by means of hydrochloric acid. In order to increase the value of the residues as fodder, the acid is freed from sulphuric acid by means of barium chloride. The sodium carbonate subsequentlyused to neutralize the acid must also be free from sulphate.

G. WASSMUS Berlin: *Manufacture of Alcohol from potatoes*, (Germ. P. 13637, Oct. 17, 1880). The saccharification is produced by means of hydrochloric acid, under a pressure of three atmospheres. The liquid is neutralized with sodium carbonate to render the residues suitable for fodder.

A. WUNSCHE, Egeln: *Preparation of Acetic Acid*, (Germ. P. 13165, May 11, 1880). The vapors containing acetic acid are passed through a rectifying apparatus, connected with a return condenser. Traces of acetic acid in the vapors leaving the apparatus are aborbed by some basic substance.

C. G. BJOERKMANN, London: *Explosive Compound*, (Eng.P. 2483, June 19, 1880). Glycerine is mixed with one third of its weight of a carbohydrate, such as glucose &c. and then nitrated. The liquid is then mixed winh combustible bodies containing oxygen, in such proportions that a powder results. It is said not to freeze and to be transportable without danger.

S. J. MACKIE, London: *Explosive compound*, (Eng. P. 4230, Oct. 18, 1880). Dry sodium nitrate is ground in a mill together with loose gun cotton. The mass is pressed in moulds, a split tube being inserted, which subsequently receives the fulminate.

JOHN PEARSON. J. WHITESTONE and R. R. GUBBINS, London: *Manufacture of Emery Wheels*, (Germ P. 12924, May 11, 1880). A cement composed of glue, tannin and methylic alcohol is mixed with powdered emery, at 105° C. and compressed in moulds.

S. JAROSLAWSKI, Berlin: *Apparatus for carrying off the foul vapors from boiling animal matters.* (Germ. P. 13426, July 29, 1880.) —A pipe from the boiler ends in a nozzle like that of an injector and within a socket connected with a supply of water. The escape of the vapors under pressure aspirates a large amount of water which condenses and absorbs the bad odors.

R. C. ANDERSON, London: *Galvanic Battery*, (Eng. P. 4267, Oct. 20, 1880.)—Composed of carbon and zinc elements. The latter are immersed in solution of magnesium chloride, the latter in potassium chloro-chromate.

H. GROUVEN, Leipsic: *Method and apparatus for manufacturing ammonium sulphate from the nitrogen contained in peat*, (Germ. P. 13718, July 1, 1880. Addition to Germ. P. 2709.)—The retorts are placed horizontally instead of vertically, as described in the original patent. Peat, mixed with lime, is heated in a series of retorts which are so connected with each other that the vapors and gases from the first one are obliged to pass through the others.

The ammonium carbonate obtained from the cooled and condensed gases is converted into sulphate by digestion with calcium sulphate. The gases from the retorts after being deprived of ammonium carbonate and carbonic acid are burned in the retort furnace.

BINDSCHEDLER & BUSCH, Basle: *Method of preparing coloring matters by the action of halogens upon the azo-derivatives of resorcin*, (Germ. P. 14622, Dec. 30, 1880.)—The azo-derivatives of resorcin are obtained by the direct azotation of resorcin by means of a saturated solution of nitrous acid in sulphuric acid or by the action of nitroso compounds of phenols upon resorcin. Bromine (etc.) derivates are prepared from the azo-compounds in alkaline solution. Upon acidifying the coloring substance is separated and can then be converted into the potassium or sodium salt. The bromine product forms a violet-blue to blue coloring matter with beautiful fluorescence.

BADISCHE ANILIN, UND SODA FABRIK, Ludwigshafen, am R.: *Method for the conversion of napthols into their corresponding primary, secondary and tertiary monamines*, (Germ. P. 14612, Feb'y 22, 1880.) By the direct action of ammonia or of substituted ammonias upon alpha and beta-napthol, alpha and beta-naphthylamine or their substitution products are formed. Of practical importance is the method for the preparation of the beta compounds, which hitherto could not be prepared on an industrial scale. For this reason also the azo-dyes of beta-naphthylamine and its derivatives have remained unknown until now. Two processes for the preparation of beta-naphthylamine from beta-naphthol are given.

W. MAJERT, Elberfeld: *Method of preparing a blue coloring matter*, (Germ. P. 14581, Aug. 3, 1880.)—If nitrosodimethylaniline in hydrochloric acid solution be allowed to react upon sulphocarbonic acid, the following reaction takes place: $3\,C_6\,H_4\,(N\,O)\,N\,(CH_3)_2 + CS_3\,H_2 = 3\,C_6\,H_4\,N\,S\,N\,(CH_3)_2 + CO_2 + H_2O$. On treating the solution of the resulting product, to which chloride of zinc and sodium chloride have been added, with an oxidizing agent, a blue coloring matter separates in flakes, whilst a red coloring matter containing sulphur remains in solution. The solution of the latter, reduced by means of zinc and hydrochloric acid until decoloration takes place, yields a further separation of the blue coloring matter upon addition of an oxidizing agent.

W. MICHAELIS, Berlin: *Method of testing cements and cement mortars*, (Germ. P. 13808, Oct. 5, 1880.)—The samples prepared in the ordinary way are subjected in steam-tight vessels to a temperature of 140° C. to 180° C. for twenty-four hours. This process shortens the time of setting and hardening of the cement considerably, so that the tests for strength, &c., can be applied immediately after taking the samples out of the apparatus and cooling them.

TH. RICHTERS, Breslau, and L. HAGEN, Magdeburg: *Purification of ammoniacal waste waters*, (Germ. P. 14210, Oct. 31, 1880.)—

The sewage, &c., is placed together with lime in closed vessels into which air is forced through a perforated pipe. The air, charged with ammonia, is passed through a Gay Lussac tower, where the ammonia is absorbed by sulphuric acid. The insoluble matters are allowed to subside in basins, passed through a filter-press, and then used for fertilizing.

L. MAUDIN and J. SCHNEIDER, Paris: *Method of removing bad smell and taste from alcohol*, (Germ. P. 13944, Nov. 9, 1880.)—Treatment with nascent hydrogen.

E. VAN HAECHT and J. SCHREINER, Drohobycz: *Method of obtaining paraffine from crude paraffine without the use of presses*, (Germ. P. 14507, Dec. 5, 1880.)—The oils are distilled off in a still by means of superheated steam until solid substances only pass over.

H. PRECHT, New Stassfurt: *Improvements in the method of separating sodium and magnesium chloride from potassium-magnesium sulphate obtained from Kainite*, (Germ. P. 13521, May 25, 1880. Addition to Germ. P. 10,637. See this Journal, Vol. II., p. 439.) A description of mechanical arrangements for separating the substances mentioned in the title as fast as they are formed, or shortly after the completion of the reaction.

L. WUESTENHAGEN, Hecklingen: *Method of and apparatus for evaporating the mother lyes in the manufacture of potash*, (Germ. P. 14015, May 1, 1881.)—The lyes, previously warmed, are run into a steam boiler for concentration, and finally evaporated in a vacuum pan to the point of crystallization. The steam from the boiler is superheated, and is used for heating the vacuum pan. The vapors from the latter are withdrawm by a pump and serve to warm the lyes.

SOCIETE ANONYME DES PRODUITS CHIMIQUES DU SUD-OUEST, Paris: *Improvements in apparatus for the manufacture of ammonia-soda*, Germ. P. 14186, Aug. 31, 1880.)—Description of apparatus for absorbing the ammonium carbonate and ammonia evolved in the treatment of the ammonium chloride solution with limestone, dolomite and, finally, milk of lime.

C. DE MONTBLANC and L. GOULARD, Paris: *Improvements in apparatus for the manufacture of ammonia-soda*, (Germ. P. 14193, Oct. 2d, 1880; addition to Germ P. 8498. See this Journal, vol. II., p. 106.)—Consist in providing the precipitating vessels with cooling jackets.

F. M. LYTE, London: *Method of obtaining lead and silver from mixed ores*, (Germ. P. 13792, Sept. 9, 1880.)—Relates to the treatment of mixed ores which cannot be easily separated on account of their nearly equal specific gravity, as galena and argentiferous lead sulphides, galena and calamine or zinc silicates, copper or iron

pyrites and zinc blende or galena, all or part of them mixed with barytes.

The general treatment consists in roasting the mixed ores to remove the greater part of the sulphur, (blendes, argentiferous lead glance and copper pyrites,) finely pulverizing and treating with hot hydrochloric acid, of 15—17%. Nearly all of the zinc and copper are thereby dissolved, whilst the greater part of the silver and lead remain undissolved in the gangue. The chlorides of silver and lead contained in the solution of zinc chloride precipitate upon cooling. By adding a fresh quantity of hydrochloric acid to the undissolved residue, nearly the whole of the lead and silver is dissolved. This solution is run into a second vessel containing a fresh quantity of the calcined ore where, by taking up zinc and copper, it becomes partially neutralized and the silver and lead chlorides are precipitated. By repeating this process the gangue, freed from zinc and copper, gradually becomes enriched in silver and lead chlorides. The latter are then extracted from the mixture by means of a hot concentrated solution of sodium chloride, from which the lead chloride separates upon cooling. The lead and silver in solution are precipitated with zinc and finally subjected to cupellation.

C. WINKLER, Freiberg: *Utilization of solutions containing metallic hyposulphates* ($R^1S\ H\ O_3$) *or thiosulphates* ($R_2\ S_2\ O_2$) *formed by the sulphurous acid contained in acid gases and vapors*, (Germ. P. 14425, Oct. 14, 1880). Obtains ferric oxide, chloride and sulphate, sulphur and sulphurous acid.

N. VON LAVROFF, St. Petersburg: *Manufacture of Phosphor-lead-bronze*, (Germ. P. 14422, Jan. 23, 1881).

H. ERNST and A. FRICKE, Goerlitz: *Apparatus for the preparation of nitrogen from air by means of substances which absorb oxygen and with the aid of a pressure of several atmospheres*, (Germ. P. 13913, Mar. 2, 1880).

I. F. ESPENSCHIED, Friedrichsfeld. *Method of preparing violet, blue and green coloring matters by means of trichlormethylesulphochloride and use of the latter for the oxidation of leuco-compounds*, (Germ. P. 14621, Dec. 28, 1880). One part of methyldiphenylamine, and one to two parts trichlormethylsulphochloride are mixed with five parts sodium chloride and gradually heated to 110° C. A copper colored melt is obtained which is successively treated with water, alkali and concentrated hydrochloric acid to remove substances which remained unacted upon. The insoluble color-base is converted by means of sulphuric acid into a product soluble in water and treated further in the usual manner. This coloring matter dyes wool and silk greenish blue. Green and violet dyes are obtained by replacing the methyldiphenylamine by ethyl or amyldiphenylamine, or by benzyldiphenylamine, dibenzylphenylamine, diphenylamine or dimethylaniline.

For the oxidation of leucobases by trichlormethylsulphochloride

one part of the former is mixed with one part of tetramethyldiamidotriphenylmethane and five parts sodium chloride and heated to 110° C. The melt is boiled with water and treated with alkali.

C. ZIEGEL, Neuwedel. *Method of tanning hides*, (Germ. P. 13920, Aug. 24, 1880.)

I. & C. BALLATSHANO and H. TREUK, Berlin. *Method of preserving and waterproofing hides*, (Germ. P. 13420, May 8, 1880).

I. C. FELLNER, Frankfort of M. *Apparatus for driving gases out of liquids arranged for continuous working*, (Germ. P. 14204, Oct. 21, 1880).

L. PERRIER, Paris: *Vapor guage for analyzing liquids and for ascertaining pressure*, (Germ. P. 13221, Aug. 21, 1880.)—This apparatus indicates the amount of alcohol contained in the vapors of stills. It consists of a pressure tube, the lower end of which is drawn out to a point which dips into the mercury contained in a small vessel soldered to the end of the tube. Above the mercury a liquid of known vapor tension is placed. From the height to which the vapor of this liquid forces the mercury in the tube the amount of alcohol in the distilling vapor may be judged.

C. R. ENGEL, Paris: *Method of preparing potassium carbonate*, (Germ. P. 15128, Jan. 29, 1881.)—Decomposes by heat potassium-magnesium carbonate, obtained by agitating magnesium carbonate with an aqueous solution of a potassium salt in presence of carbonic acid under pressure.

F. BENKER, Weinfelden: *Manufacture of sulphuric acid*, (Eng. P. 3540, Sept. 1, 1880.)—Injects the sulphurous acid into the lower part of the lead chambers in order to prevent waste of nitrate, &c.

H. BOECKEL, Stassfurt: *Method of working up Kainite and other double sulphates of potassium and magnesium with the aid of calcium chloride*, (Germ. P. 14938, Feby. 5, 1881).

C. SCHEIBLER, Berlin: *Preparation of magnesia from magnesian limestone, dolomite or calcareous magnesite*, (Germ. P. 14936, Jan. 1, 1881.)—The above mentioned substances are calcined to drive off carbonic acid, finely broken up and then treated with a 10 to 15 per cent. solution of sugar or syrup to dissolve out the lime. The solution of saccharate of lime is separated from the undissolved magnesia, &c., in a suitable manner and decomposed with carbonic acid to recover the sugar.

I. P. CLOSSON, Paris: *Improvements in the method of obtaining magnesia from calcined dolomite*, (Germ. P. 15342, Feby. 25, 1881, addition to Germ. P. 11456. See this Journal, vol. 3. p. 75.)—The decomposition of the solution of magnesium chloride by calcined

dolomite is greatly facilitated by the addition of small quantities of sugar or syrup.

B. E. R. NEWLANDS, London: *Manufacture of aluminium sulphate*, (Engl. P. 5287, Dec. 17, 1880).—Strongly presses the magma of impure aluminium sulphate to remove greater part of free sulphuric acid and soluble ferric salts.

C. HESSEL, Kilburn: *Preparation of hydrogen*, (Engl. P. 3584, Sept. 3, 1880).—A mixture of steam and hydrocarbons is injected into superheaters or retorts heated to redness. The carbonic acid formed is removed from the hydrogen by absorption.

F. X. BORSCHE, JR., Prague: *Improvements in the method of obtaining ammonia from the nitrogen contained in elution lyes, osmose waters, etc.*, (Germ. P. 14433, July 9, 1880).—The evaporated liquids are subjected to dry distillation over potash lime. The latter is obtained by moistening the carbonaceous residue of a previous operation, mixing it with slaked lime and heating.

F. J. BOLTON and J. A. WANKLYN, London: *Fertilizer*, (Engl. P. 5173, Dec. 10, 1880).—Acid calcium phosphate, extracted from commercial superphosphate, is mixed with ammonia water and the whole evaporated to dryness.

J. F. PARKER, Birmingham: *Manufacture of illuminating and heating gas*, (Engl. P. 3695, Sept. 10, 1880).—Hydrocarbons, tar oil or bitumen are mixed with 10–12 per cent. of lime, and heated in retorts. The gas is said to need no purification.

P. GREIFF, Frankfort: *Preparation of coloring matters of the rosaniline group by the action of nitrobenzyl chloride upon the salts of primary aromatic amines in the presence of oxidizing agents*, (Germ. P. 15120, Jan. 26, 1881).—A red, soluble in water, is obtained by heating to 170°–200° C. one molecule of nitrobenzyl chloride, two molecules of sulphate of aniline or toluidine and one molecule ferric chloride or other oxidizing agent.

FARBWERKE, successors to MEISTER LUCINS & BRUENING, Hoechst: *Method of preparing coloring matters from the nitro-derivatives of naphthalene*, (Germ. P. 14954, Dec. 17, 1880).—Monobrom-(Monochlor-) naphthalene is converted by energetic nitration into the tetra-nitrocompound

$$[C_{10}H_3(NO_2)_4Br.]$$

The halogen atom in the latter product can with facility be replaced by various molecular residues (hydroxyl, amidogen, etc.). From these various metallic salts of tetra-nitronaphthal are obtained which can be employed as coloring matters.

FR. GRASSLER, Cannstadt: *Diazotizing method and its continuation to the formation of coloring matters*, (Germ. P. 14950, Nov. 28, 1880.)—This process differs from the one hitherto employed, in so far as no free acid is used for the decomposition of

the nitrite. The latter is employed in the form of ammonium salt or in combination with ammonium salts. The decomposition of the mixture is produced by warming the cloth to which it has been applied.

Compagnie industrielle des procedes Raoul Pictet, Paris: Rectifying alcohol, (Engl. P. 5245, Dec. 14, 1880.)—This method is based upon the fact that the difference in volatility between the empyreumatic substances, &c., and alcohol increases with the diminution of pressure. This method is said to yield alcohol of 99%.

E. Thorn, Hamburg: *Extracting apparatus for analytical purposes,* (Germ. P. 14523, Oct. 17, 1880.)

J. Storer, Glasgow: *Apparatus for treating liquids with gases or vapors.* (Germ. P. 14900, Dec. 21, 1880.)—Within a vertical cylindrical vessel for holding the liquid a second cylinder, open at both ends, is placed axially and reaching to the surface of the liquid. A shaft studded with screw blades revolves at a rapid rate within the inner cy inder. By this means the liquid passes rapidly through it, air or other gases are aspirated into it, and the latter intimately mixed with the liquid.

Paul Casamajor and Ch. H. Senff, New York: *Improvements in filtering apparatus,* (Germ. P. 14937, Feb'y 1, 1881.) See under "American Patents," this Journal, vol. 3, p. 65.

H. Borntræger, Wurzburg: *Method of manufacturing sulphuric acid, free from arsenic and selenium, from the soda residues of Leblanc's process, with the aid of exhausted pyrites,* (Germ. P. 15757, Mar. 8, 1881.)—Sulphide of iron is reformed by digesting the finely powdered exhausted pyrites with the sulphur lyes, obtained by extracting soda waste with water under pressure,

Th. Richters, Breslau: *Method of obtaining ammonia from the purifying mass of gas works.* (Germ. P. 15206, July 31, 1880). The oxide of iron mixture as it comes out of the purifiers containsl about 0.8% NH_3, this amount is lost by volatilization during the process of regeneration of the mass as ordinarily conducted, *i.e.* exposure to the air &c. The inventor combines the recovery of this ammonia with the regeneration of the mass, by putting the latter into tanks provided with diaphragms and passing air through it. The heat resulting from the reaction drives off the ammonia which is then obtained from the exhaust air by treating the latter with acid.

A. & L. Brin, Paris: *Oxygen from atmospheric air.* (Germ. P. 15298, Feb. 2, 1881). Baryta is heated in retorts, in the usuamanner, first at a low temperature while passing air over it, then at a higher temperature to decompose the barium peroxide. The regulation of the temperature is accomplished automatically by means of pyrometers, connected with dampers which control the supply

of air admitted to the furnace. The oxgyen is exhausted from the retorts by a barometer pump, consisting of two cylinders alternately filled with water which also work automatically.

A. Baeyer, Munich: *Method of preparing paranitrobenzaldehyde* (Germ. P. 15743, Feby. 20, 1881). This substance is obtained by the limited oxidation of paranitro-cinnamic acid, or of the ethers of this acid. For this purpose the following oxidizing agents are employed: potassium permanganate in alkaline and acid solution, lead peroxide, manganic oxide and sulphuric acid, chromic acid, potassium chromate and sulphuric acid, nitric acid, or a mixture of potassium nitrate or nitric acid, and concentrated sulphuric acid.

Lembach & Schleicher, Biebrich am R: *Method of preparing coloring matters by the action of nitrobenzylchloride or bromide upon secondary and tertiary amines or phenols* (Germ. P. 14945, Aug. 21, 1880). From these reactioms which must take place in the presence of oxidizing metallic salts, blue, green, violet and red coloring matters result.

M. Prud'homme, Mulhausen : *Method of preparing coloring matters for cotton, wool and silk* (Germ. P. 15616, Jan. 28, 1881.)—From anthraquinone, alizarin, anthrapurpurin, flavopurpurin and Cœrulein nitro substitution compounds are prepared which by heating with sulphuric acid exchange the NO_2 group for the SO_3H group. These sulphonic acids, their salts and finally the hydroxyl substitution products which result from the fusion of the sulphonic acids with alkaline hydroxides are the coloring matters patented.

E. A. Schott, Kreiensen: *Manufacture of carbonized peat coal for removing coloring matters and impurities from liquids and for disinfecting atmospheric air in enclosed spaces*, (Germ P. 14923, Dec. 14, 1880).

B. J. Mills, London *Explosive compounds* (Engl. P. 800 and 801, Feb. 24, 1881) 9295 parts nitroglycerine, 5–7 parts moderately nitrated cellulose and 0.5—2 pts. alcohol. Another compound is composed of 0.5 to 3 parts moderately nitrated cellulose, 23 to 30 parts sodium nitrate, 8 to 18 parts wood cellulose and 3 to 4 parts sulphur.

H. Proudhomme, Putte Capellen : *Explosive compound* (Engl. P. 4200, Apr. 14, 1881).

C. Heinzerling, Frankfort of M *Rapid tanning process by the use of chromates or chromium salts in combination with other mineral substances and dissolved fats and with hydrocarbons*, (Germ. P 14,769 Nov. 9, 1880). Relates to the manner of applying the solutions to the hides, the modifications which can be made in the composition of the solution to suit the uses to which the leather is to be applied, and the manner of fixing the absorbed matters, &c.

ERRATA.

Pages 49, 57, 62, and 72, read Hammer in place of Hammel all through,

Page	49,	4th	line	from	top,	read Zinc for Indigo.
"	49,	14	"	"	botom,	" Employed for Examined.
"	49,	8	"	"	"	" Gallic acid for Tannic acid.
"	49,	7	"	"	"	" which for it.
"	52,	13	"	"	"	" Compensated for complicated.
"	53,	14	"	"	top,	" fine for five.
"	55,	8	"	"	"	" Tan for air.
"	56,	14	"	"	bottom,	" Insert the word Barkometer before the second word in the line.
"	56,	10	"	"	"	" Filings for Filtering.

XI. ON THE COMPARATIVE VALUE OF THE SEVERAL METHODS OF DETERMINING TANNIC ACID.

By Nelson H. Darton.

This investigation was to determine the relative accuracy of the following methods: Lœwenthal's latest modifications of his old method; Hammel's skin and specific gravity process; Carpené's indigo method as modified by Barbieri; the old gelatin process, highly refined; Allen's lead method, as modified by Sanford, and my own method, a modification of Lœwenthal's.

In the first place, I would like to state that every evidence that I can bring to bear shows that there is but one tannic acid—a principle which has the peculiar property of combining in the proportion of 10 parts by weight with 6 parts by weight of skin to form leather. In nature this principle is often mixed with glucosides, so-called extractive matter, etc., and hence the various formulæ assigned to what have been supposed to be different tannic acids. I propose to show proof of this in a subsequent paper. Where tannic acid is mentioned in this paper the above principle is meant. It can readily be obtained pure from nut galls and oak and hemlock bark. When obtained from kino and catechu (the so-called catechu tannin) it is accompanied by an extractive principle which renders the tannate of copper soluble in ammonia.

For the purpose of standardizing the numerous solutions to be used, an absolutely pure tannic acid was examined. Its preparation was conducted thus: A fair quantity of coarsely ground Aleppo nut-galls was taken and treated with ether and water, after the method of the U. S. P.

The product thus obtained was then thoroughly washed with absolute alcohol, dissolved in absolutely pure ether, and this repeatedly agitated with water until all of the tannic acid was thought to be extracted from the tannin; it was then separated from the ether by evaporation finally in a vacuum; was then dissolved in 80 % alcohol, filtered and separated from this as above; this well dried over sulphuric acid, and at a gentle heat in the air bath in a current of pure, dry carbon dioxide; a portion of it was burnt, and twice yielded a proper formula.

A solution of this was prepared containing 2 grams to a litre;

this solution was kept in a colored bottle out of contact with the air by carbon dioxide, and when not in use, for greater precaution, the bottles were placed in the dark. No change however occurred in their relation to the permanganate solution.

Pure gallic acid was prepared from nut galls. It was carefully recrystallized and readily obtained pure.

A solution of this was prepared as of the acid and was carefully kept.

Pure English gelatine (Nelson's), was used after purification, and a solution of it was prepared immediately before use by dissolving 25 grams in a litre of a 10% solution of sodium chloride.

The permanganate of potassium solution contained 1.5 grams to a litre, and was frequently renewed during the investigations, the acetate of lead solution for Allen's method contained a deci-nomal quantity of the pure acetate of lead.

The indigo carmine solution was prepared by dissolving 3 grams of the pure preparation in a litre of water. It was boiled for some time and carefully kept; did not change its relation to the permanganate solution a fraction of a cubic millimetre. The other solutions were all prepared as required with every care and precaution.

For the objects of the determinations, decoctions were prepared of hemlock, oak and chestnut barks, Aleppo nut galls, sumach leaves, and of the so-called catechu, tannins of catechu, kino and rhatany root. As they were all prepared and kept under the same conditions, I will only detail the mode of extraction of the first and most important one, hemlock. To a litre of the decoction in view 20 grams of the coarsely ground select inner bark of the hemlock was taken; this was extracted by carefully heating it with water in a current of carbon dioxide, allowing the decoction thus formed to cool, and then filtering it through four thicknesses of filtering paper; a very small amount of dilute sulphuric acid was then added to the solution and it was again filtered, the excess of acid neutralized by baryta water and filtered. The decoction was thus freed from the so-called extractive matter and contained tannic and gallic acids nearly pure. This decoction was preserved in an atmosphere of carbon dioxide. The decoctions of catechu, etc., were prepared and kept in a similar manner but still retained a strong color; they are, I may add, much less liable to change, owing to the peculiar principles present. Tannic acid prepared from these solutions by a process similar to the one used for galls, had a variable

constitution, differing materially from that of the pure tannic acid obtained from the others.

I will first consider Lœwenthal's method (Fres Zeitschr. 1877, pp. 33 and 201.) This method has been highly commended by Proctor and others, but in working with it I soon found that it gave results at variance with practical experience and with my control method as will be detailed later. Portions of each of the ten different decoctions were taken and the analysis conducted in strict accordance with Lœwenthal's directions. Thus : 20 cc. of the indigo solution was first titrated. It required exactly 8.4 cc. of permanganate solution. The titrations were conducted in a porcelain dish; $\frac{3}{4}$ of a litre of solution and 10 cc. of dilute sulphuric acid were used in every case. 10 cc. of the standard tannin solution, with 20 cc. indigo solution and acid as above, required 13.7 cc. Deducting 8.4 cc. for the indigo we have 5.3 cc. used, for the tannin.

This same operation was repeated, using the decoction instead of the standard solution. 15 3 cc. were now used, or 6.9 cc. for the decoction.

100 cc. of the decoction were then taken, placed in a flask with 50 cc. of the gelatine solution and 100 cc. of a saturated sodium chloride solution, containing also 25 cc. of sulphuric acid and 50 cc. of hydrochloric acid per litre. Between the additions the mixture was well agitated. It was allowed to stand over night, and a perfectly clear filtrate taken off the next morning. To save calculation 25 cc. of this, equaling 10 cc. of the undiluted decoction, were taken and tested as above, using 11.45 cc. of permanganate, or 3.05 cc. for the impurites, etc., not tannic acid. Subtracting this from the figure 6.9 cc. leaves 3.85 cc. and we have the equation;

5.3 : 3.85 = 0.2 grms. to a litre : x grms. to a litre.
$x = 1.45$ grms. to 20 grms. of bark, or 7.25 per cent., the value of the bark. This, as I will show beyond, is lower than is really the case. The figures obtained were:

No.	20 G Hemlock.	20 G Oak.	20 G Chestnut.	15 G N. Galls.	10 G Sumach.	10 G 5 Catechu.	2.5 G Kino.	3 G Rhatany.
1	7.25	7.40	6.00	52.5	13.5	47.0	82.0	
2	7.00	7.30	6.00	56.3	12.9	51.0	82.0	
3	7.30	7.45	6.10	54.2	12.9	48.0	82.0	
4	7.20	7.25	5.90	55.0	13.3	49.0	82.5	
5	7.25	7.00	6.30	55.5	13.1	48.0	81.3	
6	7.20	7.00	6.00	53.0	12.8	50.0	83.0	
7	6.9	7.16	5.90	55.6	13.2	51.0	82.0	
8	7.25	7.30	6.20	54.4	13.25	49.0	82.0	
9	7.10	7.20	6.00	54.5	12.95	50.0	83.0	
10	7.15	7.10	6.15	52.8	13.2	48.0	81.5	
Average.	7.16	7.21	6.15	54.4	13.1	49.0	82.1	
Real Value.	7.90	8.22	7.42	61.5	16.25	40.0	72.0	34.00

About 80.00

giving altogether an average value about 95 out of exactly 100 per cent. But as this is an average including the discordantly high and low it is not to be wondered at. The value however for the first five is about 86 per cent. and for the last two 117 per cent.

The errors are incurred because the tannate of gelatine is more or less soluble in a weak gelatine solution even when chloride of sodium or the strong acids are present; in fact although the acids facilitate coagulation they also dissolve a portion of it. It is also somewhat soluble in solutions of gallic acid as is shown by the figures in the nut gall column of the table; moreover much gelatine remains in the solution. The quantity is very variable as it depends upon the amount of so-called extractive matter present. As I will show further on all those errors together produce an effect —amount to perhaps 40 per cent. This is, however, partly complicated by the amount of oxidizable matter and gallic acid which are also, when together, precipitated partly with the tannate of gelatine by the withdrawal of the tannic acid which holds them in solution. This is shown in the column under rhatany where so much matter depending on the tannic acid for its solution was precipitated as to render even approximation impossible. It is also shown in catechu and kino but to a less extent; this is what some in good faith call tannic acid, and ascribe to it a formula. They probably never combined an ounce of tannin with skin in all their lives, so their ignorance is pardonable. In the last three determinations, I used as near as possible the exact amount of gelatine necessary and a minimum of acid solution, with but little difference in the results.

This method surely is a very satisfactory one as far as it goes, as it

is very easy to execute and has not so many of the difficulties peculiar to some of the others; it however, does not come up to my requirements. The value of this method, especially its use in determining the values of spent liquors was investigated. These liquors contain many unknown principles derived from the skins in their treatment, and thus their accurate determination is very difficult. This and another method of determination are the only ones that can be used at all. It is of great importance to the tanner to know how his spent liquors and the spent barks run in strength. The spent liquors were taken, treated with sulphuric acid, etc., as above, and the determination proceeded with. Five analyses were made of the spent liquors direct from the vats after a run with Buenos Ayres skins well cleaned and raised—oak and hemlock liquors and those from catechu were also taken. They had been used on five skins but were good samples. The results are stated in absolute per cents.

No.	Oak.	Hemlock.	Sumach.	Catechu.
1	.13 per cent	.08 per cent	2.95 per cent	2.90 per cent
2	.14 per cent	.085 per cent	2.90 per cent	2.93 per cent
3	.125 per cent	.08 per cent	2.95 per cent	2.93 per cent
4	.16 per cent	.08 per cent	2.93 per cent	2.89 per cent
5	.145 per cent	.085 per cent	2.90 per cent	2.90 per cent
Aver.	.140 per cent	.0825 per cent	2.93 per cent	2.91 per cent
Real value	.27 per cent	.21 per cent	1.62 per cent	1.90 per cent

These figures are, of course, only approximate, but are quite exact proportionally. The causes of such great errors are these: Much less matter is separated with the tannate of gelatine, a very large proportion in fact, nearly all of the latter dissolves and the amount of gelatin left in solution is very considerable, so that there is merely a perceptible difference in each two determinations. With spent barks there is but little difference from the result noticed above, they being a little higher by compensation.

Now the prime argument. To show the misleading results given by this method and to show the control used, we will consider hemlock bark No. 1 analysis all through: Here in the first table we find it to contain 7.25 per cent. of tannic acid. We take a cord of this bark, leach it, and obtain 1,250 gallons of decoction. The spent bark is analyzed by this method, and found to contain .7 per cent. of tannic acid. Leather is tanned from the decoction with great precaution, and what is 5 per cent. of the bark found to have been used. As I will show further on, this was pure

tannic acid. The spent liquor is also examined, and found to hold what was equivalent to 5 per cent. of the bark in tannic acid. That is to say, the same—as we would speak of the spent liquors as containing nearly all the tannin of the bark, or 6.5 or 7 per cent. of it, in tannic acid. However, all these figures added together give a result of 6.2 per cent., or 1 per cent. unaccounted for, thus showing the result to be too low. The tannic acid had not decomposed, as I used a preservative to keep it, and one which I know to be thorough and complete.

Now, if the spent liquors be examined by say the method I will note further on, we find it to contain just twice as much, or 1.4 per cent. This would bring it up to 6.9 per cent.; still too low; but by examining the spent bark in the same process we have 1.5, instead of 5, and the figure is brought up to its nominal value then proved of 7.9 per cent.

According to the reasoning deduced from this process, we would have 6 per cent. of the bark utilized as tannin to combine with the leather. Nothing is more erroneous than this, as is proved every day by the tanner; as never over 5 per cent. is obtained from hemlock, the others in close proportion. So much for this process.

Next in order of our consideration comes Hammond's method, and its modifications, which were so popular some time ago; among some of the tanners the results obtained by it were so inaccurate and inconsistent as to speedily set it aside. The many inaccuracies have been partially shown by a number of investigators, especially Proctor, who has shown that gallic acid is thrown down with the tannin, etc. The method was as carefully tested and examined as the other, and the results are here tabulated :

No.	Hemlock.	Oak.	Chestnut.	Nut Galls.	[Sumach.	
1	10.2	13.2	10.0	34.0	10.00	Catechu,
2	6.5	6.4	4.8	43.0	11.00	
3	9.9	9.3	9.2	56.0	6.5	Kino, and
4	11.4	12.4	7.4	40.0	7.6	Rhatany,
5	9.6	14.0	6.9	61.0	9.8	
6	6.8	4.0	11.2	52.0	6.9	not ac-
7	11.2	8.0	10.2	57.0	8.2	cordant
8	6.9	12.5	4.9	49.5	7.4	
9	8.4	5.0	8.6	45.0	10.5	results.
10	10.5	10.0	9.4	56.0	6.8	
Average,	9.14	9.48	8 26	49.0	8.57	
Real value,	7.90	8.22	7.42	61.5	16.25	

These figures were obtained in the following manner. The decoctions, filtered and treated as before, were placed in a graduated jar, and the specific gravity taken by the use of a very delicate densimeter. The skin was then taken, thoroughly cleansed, heated and raised by appropriate treatment. It was then thoroughly washed with a solution of gallic acid, again with acetic acid and warm water, until it was pure and sweet. It was then placed in water with the decoction, and allowed to air for an indefinite period, for the first five analyses. It was then taken out and scrubbed off as far as possible without altering the bulk of the solution, the specific gravity of which was then taken. The amount of tannic acid was calculated from the difference, etc.

The details of the method were executed with much more refinement than is the case generally, and the principles involved thoroughly worked out. But with this look at the result one might reason, Why would not the result be identical with the practical manufacture of the leather, or 5 per cent, etc? This I can readily show. In the first place, to obtain the decoction for their determination, or rather to make solutions uniting all the tannin in the material used, we have to extract much more extraneous matter, especially in the case of rhatany, kino and catechu, than in the imperfect leaching of the tanneries, where in the case of the last materials only 50 per cent. of the tannin present, and a minimum of coloring matter, are extracted; thus the the leather has less to contaminate it. In the second place the sample of skin is never a fair specimen in the treatment of a whole hide, and besides, the leather formed cannot be properly freed from the different matters besides tannic acid soaked into it, without altering the volume of the solution. It must be remembered that leather after it is formed is thoroughly rolled, and scrubbed so as to leave nothing but leather. The leather absorbs tannic acid and the so-called extractive matters; and by withdrawing the tannic and partly the gallic acid from a solution much matter is deposited, partly in the solution, some in the leather. This by the above treatment is removed before the leather is dried and weighed; thus much matter is abstracted and accounted for as tannic acid, this yielding too high results. Besides these considerations the densimeter must have extreme delicacy, and even then a small space on the scale represents much tannin. Altogether, I pronounce this method little better than a guess; in fact a tanner can arrive at the strength of his liquors by their taste better than by this method.

In the examination of spent liquors and barks this method was of no value at all, as it gives such very inconsistent results. This is caused by the putrescent matters present in the liquor and other causes. In the spent liquor the analyses turn out like those shown in the table below :

No.	Oak.	Hemlock.	Sumach.	Catechu.
1	6.5	4.0	7.9	4.0
2	2.2	5.2	8.4	3.0
Average,	4.3	4.8	8.2	3.5
Real value,	.27	.21	1.62	1.90

In fact, Hammel's whole method, however modified, is the same thing. This instrument should be done away with in the tanneries as it only shows how much matter, tannic and otherwise, especially otherwise, *is in solution.*

In the use of this method and the worthless modification of drying the skin filterings and weighing before and after treatment, chemists and others generally obtain the results too low, but that depends on the very nice point of understanding how to make leather.

Using the calculations applied to Lœwenthal's process for a control we find thus: In No. 1 the leather 5 per cent., the spent liquor 3.5 per cent., the spent bark 4.5, all together 13 per cent. This is quite far from the average value of the bark, which will be noticed was 9.48. Giving the proper value to the spent liquor (1.4 per cent) the result is 10.9, or much nearer the truth.

PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

Meeting held May 5th, 1882.

The meeting was called to order at 8:40 p. m. Prof. A. R. Leeds in the chair.

The minutes of the previous meeting were read and approved.

The report of the Board of Directors was read and accepted.

Dr. Doremus on behalf of the Committee on papers and publications stated that he was able to present to the society this evening, all the proceedings, and nearly all the papers read before the society from January 1st up to date.

He further stated that the next number of the Journal would be out by May 25th. He also said that the Committee are in need of more abstractors and earnestly hoped that the society would support them in this direction.

As there are from three to four fly leaves of the Journal that can be devoted to advertising, the Committee thought that the money that could be obtained from that source would be a great help to the Journal.

The present issue of the Journal would cost about ninety dollars, the number of pages being somewhat larger than was originally intended. But he thought that if the Committee could count upon publishing a number of the Journal of thirty-two pages each month, that the cost would be reduced to sixty dollars per month, or about five hundred and fifty dollars for the year, including the ninety dollars for the present month.

The treasurer stated that money was coming in fast at present.

The librarian had nothing to report except that a number of periodicals had come in.

The following gentlemen were nominated :

G. Polledo, as associate member. Proposed by T. O'C. Sloane, James H. Stebbins, Jr. and Arthur H. Elliott.

H. Edward Stockbridge, as regular member, by C. A. Goessmann, H. Endemann and James H. Stebbins, Jr.

Dr. Wm. Strattford, as regular member, by Charles A. Doremus, R. A. Witthaus and James H. Stebbins, Jr.

E. J. Mallett, Jr., as regular member, by Charles A. Doremus, James H. Stebbins, Jr. and A. R. Leeds.

After which the first paper of the evening, on Acrolein-urea, was read by Dr. A. R. Leeds. After some questions by Mr. Elliott, the second paper of the evening. Note on an earthy ferric sulphate, by Charles E. Wait followed. Mr. Darton's paper on the determination of tannic acid was then read by title.

The following papers were announced for the next meeting:

1st. Use of the tannometer in tan yards, by Nelson H. Darton.

2nd. On the determination of phosphorus in iron ore, by E. Waller.

3rd. Report on the progress of industrial chemistry, by Dr. E. E. Brenneman.

Dr. Waller moved that when the meeting adjourns, it shall be adjourned to the next conversazione. Seconded and carried.

Dr. Doremus moved that all papers read before the society shall become the lawful property of the society till thirty days from the date of reading. After a number of suggestions by different gentlemen, Dr. Doremus' motion was carried. The meeting then adjourned.

JAMES H. STEBBINS, Jr.,
Recording Secretary.

XII. ACROLEIN-UREA.

BY ALBERT R. LEEDS.

In the *Annalen der Chemie und Pharmacie* for 1869, (vol. lxxv p. 203), Hugo Schiff has described, under the name of acrylureid, a condensation product formed by the union of two molecules of urea with one of acrolein, in accordance with the reaction:

$$2\,[CO.\,(NH_2)_2] + C_3H_4O = \left.\begin{matrix} CO.\,N_2H_3 \\ CO.\,N_2H_3 \end{matrix}\right\} C_3H_4 + H_2O$$

Without being aware of Schiff's labors, and whilst studying the aldehyde compounds with quite different objects than those followed by Schiff, I obtained the same compound, but had assigned to it a quite different formula. The explanation of these differences becomes apparent on studying the method by which Schiff prepared the compound, since this method did not and could not yield the substance in question. He mixed a concentrated aqueous solution of urea with acrolein, and after his acrylureid had gradually precipitated as small while needles (?), he washed it merely with ether and water, and dried *in vacuo*. He likewise states that powdered

urea unites with acrolein directly. "The mixture swells up, and is converted into a white brittle mass, which in some places has an entirely crystalline appearance. By far the greatest portion is intimately mixed with a white porcelain-like product, formed by decomposition of the acrolein." The analysis of this body gave, as might readily have been expected, no satisfactory results. Schiff further states that in various preparations, some of which were prepared at ordinary and others at higher temperatures, he obtained carbon varying between 44 and 47 per cent., and hydrogen between 6.9 and 7.1 per cent. The formula of diacryltriureid requires 42.2 per cent. carbon and 6.3 per cent. hydrogen. That of acryldiureid, which Schiff adopted, but 38 per cent. carbon and 6.3 per cent. hydrogen.

Throughout his investigation Schiff worked upon impure substances, and in that way obtained not only varying results, but results which would support no rational formula, the one which he finally adopted,—that of diacryltriureid, being altogether erroneous.

In the first experiment made by the author, the acrolein obtained by heating anhydrous glycerin with potassic bisulphate was allowed to distil over directly into an alcoholic solution of urea. A precipitate formed at once, and after a time a considerable amount was obtained, the urea however being still in excess. The filtrate was of a reddish color, and on evaporation yielded a resinous or gummy mass, which could neither be sublimed or crystallized from solvents. The precipitate left on the filter was of a yellow-white color, but after repeated treatment with alcohol became quite white, and lost all of the gummy or thick oily substance at first adhering to it.

Its analysis showed that it had been formed by the union, not of two, but of one molecule of urea, with one of acrolein, a molecule of water being eliminated, and the resulting compound having the formula $C_4H_6N_2O$ or $CO(NH)_2C_3H_4$.

	Found.	Theory.
Carbon	49.01	48.98
Hydrogen	6.3	6.12

Not only as originally prepared by repeated purification with alcohol, but also after treatment with carbon disulphide, chloroform and ether, in all of which it is only slightly soluble, every attempt to obtain it in a crystallized condition was unsuccessful. It did not melt nor sublime, and when strongly heated underwent decom-

position. It was readily attracted by nitric acid, but the products of the decomposition were not studied. From its solution in alcohol no precipitate was obtained on the addition of bromine. On treatment with hydrochloric, tartaric and acetic acids no salts were formed.

The alcoholic extracts were collected, and yielded, after successive solutions in alcohol and precipitation with water, a white substance similar to that above described, and having a melting-point of 185°. Since sufficient of this material could not be obtained to admit of its satisfactory purification and analysis, a much larger amount of urea in alcoholic solution than that used in the foregoing experiments (40 grms.) was treated with acrolein in successive portions in a closed vessel, until after long continued warming the odor of acrolein ceased to disappear. In this case, unlike the foregoing, no precipitate was formed, nor did any take place until after the addition of water, when a very abundant white amorphous substance was thrown down. This was boiled with alcohol a great many times, each extract affording a white precipitate when diluted with water, and the portion of pure material obtained eventually was not more than that procured in the first instance, when but a small amount of urea was used and the acrolein was not added in excess. The composition of the substance thus obtained was $CO.(NH)_2.C_3H_4$, and it was identical in all respects with the substance formed in the first case. From the alcoholic extracts no other substance could be obtained, and the explanation of the small yield of the pure acrolein-urea in the second case, was eventually found to be due to the presence of the oily or gummy substance, formed as a by-product. The amount of this by-product was apparently much greater in the second case, and explained the non-precipitation of the acrolein-urea until after the addition of water to the original solution of the urea in alcohol. And the tenacity with which it adhered to the acrolein-urea on precipitation with water, and carried it into solution again on each successive boiling with alcohol, explains the very small eventual yield of the pure material. It was also due to the small adherent traces of this oil, that the second body, the one soluble in alcohol, had a melting point of 185°, and was supposed to be something else than the acrolein-urea.

The presence of this oil, which I was unable to isolate, explains all the above difficulties and the erroneous results obtained by Schiff. In conclusion, it may be safely stated that besides the small amount of this oily by-product, the only compound formed by the

action of acrolein on urea, was that containing the residue from one molecule of urea, the compound $CO.(NH)_2.C_3H_4$.

XIII. NOTE ON AN EARTHY FERRIC SULPHATE FROM ARKANSAS.

By Chas. E. Wait.

Several weeks ago a sample of earth was sent to me for examination, from Southern Arkansas. It is a friable, yellowish substance, partly soluble in water, streak uncolored, taste astringent.

Upon analysis I find it contains the following:

	PER CENT.
Insoluble in cold water	12.61
Soluble in cold water	87.39
	100.00

The soluble portion consists of

Fe_2O_3	21.82
SO_3	33.81
H_2O	31.76
	87.39

By assuming the following arrangement,

Fe_2O_3	21.82
SO_3	33.81
H_2O combined	22.41
H_2O hygroscopic	9.35
	87.39

we are able to deduce a formula consistent with that of the native persulphate of iron, viz.: $Fe_2(SO_4)_3(H_2O)_9$.

If all the water be considered as belonging to the compound, the formula will be $Fe_2(SO_4)_3(H_2O)_{13}$.

It is said an unlimited supply of this substance may be obtained.

XIV. ON THE COMPARATIVE VALUE OF THE SEVERAL METHODS OF DETERMINING TANNIC ACID.—PART 2.

By Nelson H. Darton.

The next method examined was that of Carpené, as improved and modified by Barbieri. This method was looked forward to as an excellent one when first proposed, and consequently was brought into use almost immediately. The details of the execution were very simple, and its originators obtained excellent results. Some time after, Kathreiner examined into its value, and stated* that with it he had only been able to obtain exceedingly inaccurate and discordant results, and had failed in his endeavor to favorably modify it. In his investigations he employed a decoction of sumach and a solution of the so-called "pure tannin" of the shops, and thus the comparison between the sumach and this compound, containing not over 80 per cent. of digallic acid, was not a correct one. The other 20 per cent. of the so-called "tannin" was composed, as I have found, of matter nearly all precipitable by ammonia solution, and the importance of taking this into consideration is shown below. Thus the poor results obtained by Kathreiner are explained. I have, in the investigation of this subject, applied this method to most of the other tanning materials, and have in this manner brought some new facts in relation to it to light, not only by employing the variety of the substances tested, but also by varying the conditions of procedure and investigating the source of the errors. My general mode of procedure was as follows: Using as before, for example, the decoction of hemlock bark, though not in strict accordance with the details as published by Bolley in his *Handbuch*, but with as much modification as experience had shown to be desirable. The decoction was taken in convenient quantity, an excess of a solution of ammonia added to it, and the precipitate thus produced filtered out. A known amount, which was also an excess, of a solution of ammonio-acetate of zinc was then added, and the bulk of the mixture brought up to a given point by the addition of sufficient water; the precipitate formed was allowed to separate as far as possible by standing, and the mixture then boiled down at a low heat in a partial vacuum to half (or to any specified degree) of its original volume. After allowing it to cool thoroughly the

* Dingler's Polyt. Journal 227, p. 481.

precipitate was filtered out and washed as completely as possible with a known volume of water heated to about 80° C.; it was then dissolved in warm diluted sulphuric acid, titrated with the permanganate of potassium in presence of indigo-carmine,

No.	Hemlock.	Oak.	Chestnut.	Nut Galls.	Sumach.	
1	9.30	10.00	5.50	67.00	10.00	Catechu,
2	12.00	12.50	6.50	69.00	13.50	Kino
3	10.00	9.50	5.20	65.50	11.00	and
4	11.00	11.20	7.00	68.20	13.00	Rhatany,
5	8.50	9.00	6.00	60.00	9.00	
6	18.60	13.00	9.00	69.00	19.00	non-
7	11.00	12.00	6.00	66.00	11.00	accordant
8	20.30	15.00	11.00	72.00	20.00	results.
9	8.50	7.50	6.20	60.00	14.50	
10	3.00	5.20	2.80	47.50	3.50	
Average.	12.20	10.40	6.50	62.40	12.50	
Real value.	7.90	8.22	7.42	61.50	16.25	

and the result calculated from the data so obtained as in Lowenthal's method. Above is a table of the results obtained, in percentages. The great differences which it will be noticed, occur so abruptly are principally caused by varying the conditions, and I will detail them individually. Result number one was obtained by having the amount of zinc solution, in excess, a minimum, and conducting the evaporation in the shortest possible time. Number two was obtained by omitting the preliminary ammonia treatment preceding the addition of the ammonio-zinc solution, and in exact accordance with the detail of the method as originally proposed and published in *Bolley's Handbuch*. It is the treatment with the ammonia that causes the great difference in these two results, as a fair amount of precipitate was in that way separated which would otherwise have been precipitated with the tannate of zinc by the ammonia then added. Result number three was obtained by titrating the solution before and after the separation of the tannic acid by the zinc under the same condition as in number one, thus the method was modified to the principle involved in Lœwenthal's. Result number four was obtained in the same manner, except with the omission of the ammonia treatment. Notice the difference in the result. Returning now to the original mode, in number five the evaporation of the mixture was to only two-thirds of its volume, and in number six to one-third ; numbers seven and eight were

respectively conducted under the same conditions as numbers five and six omitting the preliminary ammonia treatment. Two blank samples of decoction were taken in this connection and evaporated down to one-third of their original volume, one after the preliminary treatment with ammonia the other omitting it. In both cases considerable matter deposited, although more in the latter than in the former, thus showing how numbers six and eight have been influenced. Result number nine was obtained by using almost the exact amount of zinc solution necessary, exaporating at a very moderate rate down to two-thirds, and with the preliminary ammonia treatment. Number ten was obtained by just the reverse of number nine, with the exception that the evaporation, although very rapid, was down to the same point. All the figures together have a value of 85% ; result number nine a value of about 82%, higher by far than any single one of the other results, but still too low for accuracy, as a confirmatory duplicate of this result yielded a value differing by 10% at least. The causes of the discrepancies are these: The compound formed with the zinc is quite soluble in an excess of zinc solution; this was ascertained by several trials. Besides this error, which in number ten is shown to be considerable, in the concentration of the mixture other matter precipitates mixed with the zinc compound besides that which would be thrown down by the preliminary ammonia treatment, although I have found that the matter which has the tendency to precipitate when the tannic acid, its solvent, is abstracted from the solution, is held in solution by the free ammonia present, and thus in this and my own method that source of error is not introduced to the slightest extent, while, as I showed before, it so markedly influences the results of Hammel's and Lœwenthal's method. In boiling the mixture down at too high a heat the ammonia is evolved and this matter besides the gallate of zinc, etc. precipitates for want of the solvent. This is well shown by the lower result of number nine, which, as I before noticed, was evaporated more slowly than number one.

Take this method and modify it by the preliminary treatment with ammonia and filtration, which is shown by the above reasoning to be of such importance, use little or no excess of ammonio-zinc solution, by finding the quantity necessary by a preliminary treatment, evaporate the mixture down by one-third of the original volume at a moderate rate, and thus results similar to number nine may be obtained. I will notice here that the tannate of zinc formed in this process, as well as the tannate of copper, as I have shown in a pre-

vious paper, are quite free from extraneous matter when from the oak, hemlock, galls, etc., but from the catechu, kino, etc., are very markedly contaminated, and thus the notice in the last column. As the examinations in this line were not continued, on ascertaining the first few results, with spent barks and liquors this method yielded too unsatisfactory results to be used at all and thus I am unable to tabulate my conclusions as I did of Lœwenthal's and Hammel's methods, but I might add that it compares favorably with either in all its relations.

The next method examined is the gravimetric gelatin method, which is objectionable as not being a volumetric one. When first proposed by Sir Humphrey Davy this method was much used, and is in fact up to this day in more or less constant use in the primitive condition; but after Watts published his researches upon the numerous inaccuracies, showing that the tannate of gelatin was exceedingly soluble in an excess of its precipitant, taken together with the difficulty encountered in filtering it off, owing to its slimy nature, it lost favor, and was seldom used by scientists. Some time ago Stoddart showed that by adding alum to the solution, both of these difficulties might be avoided to a great extent and fair results obtained, although if not of the absolute amount of tannic acid present, between this and the amount available for the formation of leather. But taken under any condition, this method, as I will show, cannot be compared with Hammel's, or much less Lœwenthal's. In the examination of the method a series of five determinations each were made, and the results shown by the table below in percentages. All the causes of these grave errors, excepting the one I will notice below, are of the same character as those I detailed under Hammel's method, but without the introduction of extraneous matter, as when using hide. They have acted to a much more limited extent in this method, however, and thus the difference in the results.

	No.	Hemlock.	Oak.	Chestnut.	Nut Galls.	Sumach.	
	1.	5.30	6.20	3.50	50.20	10.00	Kino,
	2.	4.20	3.80	3.60	52.00	13.00	Catechu and
	3.	6.00	5.50	4.90	54.00	11.00	Rhatany, non-
	4.	3.70	4.50	6.40	51.50	9.50	accordant re-
	5.	4.50	4.30	5.30	56.00	14.00	sults.
Average,		4.70	4.80	4.70	52.90	11.50	
Real value,		7.90	8.22	7.42	61.50	16.25	

Owing to the influence of a cause comparatively absent in Hammel's method, the results are not solely affected by these inaccura-

cies. This cause is the solubility of tannate of gelatin, even in presence of the acids of Lœwenthal's method, though a little less there if alum has acted to such a degree as to make the results as low as some of the lower ones of Hammel's method.

Besides this, it may be well to recall to your attention here, that a solution of gallic acid is a powerful solvent, and especially in relation to this precipitate. The figures for the table were obtained in the following manner: The decoction was taken in measured quantity, mixed with sufficient alum solution and filtered from the resulting slight precipitate. A trial precipitation was first made to ascertain the approximate quantity of gelatin solution necessary, and then to another portion the necessary amount was added. The mixture, after a violent agitation, is set aside over night, and upon the next morning the precipitate is filtered off and washed with sufficient water at 5°C. After draining over the pump it is dried at 90°C. and weighed in the filter paper, the weight of which is known. The results in the table show this method to yield entirely too variable, and at the same time low figures, and therefore that the method cannot be employed in any determination where accuracy and speed are sought. The table shows a value of 87 %, I may add, but varies from this to 30 %. With "spent liquors" and "barks" the results are still more erroneous. Below are five determinations of the value of "spent liquors" in percentages of their weight:

No.	Hemlock.		Oak.	Sumach.	Catechu.
1	Hardly determinable.		0.10	.90	.50
2	"	"	0.10	.40	.60
3	"	"	0.10	.70	.50
4	"	"	0.10	.50	.70
5	"	"	0.10	.60	.60
Average,			0.10	.62	.60
Real value,	0.21		0.27	1.62	1.90

These figures do not afford me the necessary data to reason from in relation to the control, which is in this method thus unattainable.

The next method examined was the volumetric modification of the above. This method, which is even in use at this time, is nearly as inaccurate, and although a volumetric method, even as tedious as the other. The principal difficulty in the execution, and also in the accuracy of this method, is distinguishing the point at which there is no excess of either gelatin or tannic acid. This is literally impossible, as in the titration we encounter a condition in which

either the one or the other will produce a marked opalescence, and this lasts until a cubic centimetre of the one has been added in excess. This in itself condemns the method, besides there are the same inaccuracies which influence the results of the first and last method discussed. Another thing to be considered in both this and the preceding methods is, that tannic acid combines with a proportion of gelatin depending upon the amount of tannic acid present, or rather remaining in solution. To express it simply, the first portion of tannate of gelatin precipitating contains a much greater proportion of tannic acid than that falling when the quantity of the latter has decreased. Thus very variable amounts of the titrating solution may be used under varying degrees of dilution, and less or greater additions of it. In obtaining the results shown below in the table, the volumes of the standardizing solutions and those under examination were kept equal, as was also their approximate strength. The gelatin solution was added in c. c.'s at a time, and thus similar conditions existed as far as possible. The first five results were obtained from comparison with a standard solution of tannic acid, containing one gramme to a litre, and the others with the regular solution prescribed before for the other determinations. The mode of procedure was essentially as follows: The decoction in convenient quantity was placed in a beaker standing upon a blackened plate, and the gelatin solution added from the burrette in c.c.'s at a time, at first at intervals of ten minutes, then of twenty minutes, and finally of one hour; when the end reaction was approximately found by taking out drops on a glass plate placed over some black glazed paper:

No.	Hemlock.	Oak.	Chestnut.	Sumach.	Nut Galls.	Catechu.
1	3.50	4.00	3.50	10.00	49.00	25.00
2	7.50	6.00	3.90	8.00	54.00	31.00
3	6.20	3.40	6.20	11.00	57.00	17.50
4	4.00	5.50	4.50	9.50	51.00	32.20
5	5.00	4.50	5.50	3.00	53.00	35.00
6	6.40	7.10	4.60	11.00	52.00	29.00
7	4.20	5.50	5.40	13.00	56.00	34.00
8	3.90	6.30	6.40	9.50	53.00	33.00
9	4.70	4.20	3.80	11.20	51.90	34.50
10	5.20	7.30	4.70	11.60	54.00	31.50
Average	5.10	5.43	4.85	10.78	53.20	30.27
Real value.	7.90	8.22	7.42	16.25	61.50	40.00

The average value thus deduced is 84 per cent., a little less than that of the others, but the figures are so discordant they condemn the method as little more than a mere surmise. In the determination of the strength of "spent barks" and "liquors" the results obtained are even of less value, as the following table will show: Numbers in percentage:

No.	Hemlock.	Oak.	Sumach.	Catechu.
1	trace	.15	.90	.80
2	.10	.20	.50	.90
3	trace	trace	.70	.80
4	.10	.10	.90	.70
5	.15	.15	.80	.90
Average,	.07	.12	.76	.82
Real value,	.21	.27	1.62	1.90

These figures were obtained as in the first five in the preceding table.

The next method examined was that of A. H. Allen, or rather Stoddart's modification of it. In the examination of this method the first five series of results were obtained by using swollen gelatine to separate the tannic acid, and in the last five by using a piece of hide properly cleansed, bated and raised. The general mode of procedure was as follows: A convenient amount of the decoction is measured out, and the standard solution of acetate of lead added, until a drop of the mixture filtered off ceases to color a mixture of ferricyanide of potassium and solution of ammonia with the characteristic red tint peculiar to the presence of, or in this case the excess of, tannic acid. Another portion of the decoction is then taken and the tannic acid separated from it as far as possible by the hide or swollen gelatin, as the case may be, and the remainder titrated as before. If the method was as accurate as might be supposed, no tannic acid would remain in the solution after this treatment, to afford the end reaction necesary in this titration, but as sufficient tannic acid does remain the end reaction is readily found. If the absorption of the tannic acid by the hide or gelatin was not so extremely tedious, and the errors noticed under Hammel's method more or less introduced, this method would rank far above Lœwenthal's, as the end reaction is perfectly perceptible when only $\frac{1}{1000}$ part of tannic acid is present; and besides this, it is somewhat unsatisfactory to be necessitated to filter off small portions continually for observing the end

reaction. The results shown in the table below are very fair, but too high in the first five; and altogether unsatisfactory in the others.

Number.	Hemlock.	Oak.	Chestnut.	Nut Galls.	Sumach.	Catechu.	Kino.	Rhatany.
1	9.50	10.00	9.00	69.00	19.50	47.00	82.00	36.50
2	9.50	10.00	9.50	68.00	21.40	44.00	76.00	39.00
3	9.80	10.00	8.50	69.00	.....	.....	74.00	.....
4	9.60	11.00	10.00	67.00	.. ..	. ...	.....	.....
5	9.40	10.50	9.00	67.50	.....	.....	.....	.....
6	7.10	6.50	11.00	52.00	14.30	51.00	64.00	42.00
7	11.60	9.30	6.00	67.00	21.00	38.00	.....	29.50
8	12.60	6.90	4.30	49.90	19.00	.....	.....	.....
9	6.40	8.40	9.20	57.00	.....	.....	.....	.....
10	9.20	11.10	7.00	67.50	.....	.....	.....	.....
Average,	9.37	9.37	8.35	63.30	19.10	45.00	74.00	37.00
Real value,	7.90	8.22	7.42	61.50	16.25	40.00	72.00	34.00

The value deduced from these figures is about 90 %. With "spent liquors" and "barks" the results obtained are even more fluctuating. Below is a table of a few determinations of "spent liquors;

No.	Hemlock.	Oak.	Sumach.	Catechu
1	1.50	2.00	4.00	3.50
2	1.50	2.50	4.50	3.50
3	2.00	2.00	3.75	3.50
4	1.25	2.00	4.00	....
5	2.00	2.25	4.00	....
Average,	1.65	2.15	4.50	3.50
Real value,	0.21	0.27	1.62	1.90

in these the tannic acid was separated by gelatin, and these although more at variance with the truth than the others, are of some slight value when compared with Hammel's method. If in this method the tannic acid could be separated by some more appropriate process, it would yield even better results than any other now known. The next methods examined were those of Clarke and of Jean. The first is an ingenious method, using, as we know, a standard solution of cinchonia sulphate and magenta for an indicator, although yielding extremely poor results as at present executed. In fact, I saw almost immediately that nothing could be learned by examining it further than to justify these remarks. I believe that it is capable of favorable modification, and I will attempt to do this if possible. Of the last, Jean's, I can say but little; it, as some may know, consists of titrating with iodine, using starch as an indicator. The results obtained in a few preliminary

examinations were sufficient to discourage further investigation. It did not yield a value of over 60 per cent. Besides an error in the principle involved, an error is present similar to the principal one in Allen's method, that is, in the separation of the tannic acid by hide, etc., between the titrations of which six at least are necessary, even after the solutions are standardized (which also requires six), the first two for the approximate value, and the four others for the two final determinations.

To Mettenzway and Terriel we are indebted for the most ingenious as well as purely scientific method of determination, and one yielding far more accurate results than any which Proctor has examined. The method, I may add, consists in the deduction of the amount of tannic acid present by the amount of oxygen it is capable of utilizing to become oxidized. This method, however, has the insuperable disadvantage connected with it of having a very difficult mode of procedure, and is only successful under the careful manipulation of experienced hands, thus rendering it impossible except under special circumstances. Some time ago, but not in view of these investigations, I examined into its accuracy and formed these conclusions. In time I propose to modify this method if possible, to render it more simple in detail.

The next and last method examined was that of my own modification of Lœwenthal's and of Fleck's methods which I proposed some time ago before this society. I have given this method a most thorough and prolonged series of examinations, carefully controlling the results with working tests on a large scale. As it would take up too much space to detail the entire result of my investigations upon this method, I will only give a portion of them fairly and impartially selected, and at the same time from those executed under the the same conditions as were the preceding examinations. I will not detail my mode of procedure, as I have discussed it before the society at previous meetings, but I will here remark that the preliminary treatment of the decoction with diluted sulphuric acid is absolutely necessary, as the ellagic acid thus separated would otherwise interfere seriously with the copper precipitation, and would also call attention to the necessity of a preliminary ammonia treatment and filtration as in Carpené's method. In relation to the titration the suggestion of Proctor is available here. He suggests that the titration be conducted in a white porcelain dish and the first occurrence of the red or pink tint be noted, rather than

the yellow which is looked for when the titration is conducted in a flask. This red or pink tint is formed by the addition of one drop of the permanganate solution in excess and is observed around the edge of the apparently colorless space, where the depth of the solution is slight, next to the porcelain on the sides of the dish. I also wish to call attention to the necessity of using twice as much permanganate for the solution of indigo carmine alone, as is necessary for the tannic acid, etc. In the solution or decoction, as the case may be, I prefer to obtain my figure not by comparison with oxalic acid but under corresponding conditions with absolutely pure tannic acid.

No.	Hemlock.	Oak.	Chestnut.	Nut Galls.	Sumach.	Kino.	Catechu.	Rhatany.
1	7.80	8.10	7.50	59.50	17.00	86.00	45.00	57.00
2	7.85	8.10	7.50	59.00	16.50	84.00	45.00	55.50
3	7.75	8.15	7.50	58.50	16.90	86.00	45.00	58.00
4	7.95	8.25	7.45	59.00	16.75	85.00	45.00	57.00
5	8.05	8.20	7.50	59.00	16.40	86.00	45.00	59.00
6	7.85	8.10	7.40	59.00	15.95	84.00	44.50	56.00
7	7.80	8.30	7.35	60.00	16.20	85.00	46.00	56.50
8	7.90	8.25	7.45	59.00	17.00	85.00	45.00	55.00
9	7.75	8.10	7.40	60.50	16.50	84.00	45.00	54.00
10	7.70	8.20	7.50	59.50	16.50	86.00	46.00	56.50
Average,	7.83	8.15	7.45	59.20	16.57	85.20	45.15	56.70
Control or real value,	7.90	8.22	7.42	61.5	16.25	72.00	40.00	34.00

I will not make any remarks on this table except that it has a comparative value of 95% altogether, or 97% in the first five series. With "spent liquors" and "barks" the results are equally satisfactory.

No.	Hemlock.	Oak.	Sumach.	Catechu.	Hemlock.	Oak.
1	.20	.250	1.50	1.95	1.50	.90
2	.20	.260	1.50	2.00	1.40	.95
3	.19	.250	1.50	2.00	1.50	.90
4	.21	.280	1.55	1.90	1.50	.90
5	.20	.250	1.50	1.95	1.40	.90
Average,	.20	.255	1.51	1.96	1.46	.91
Real value by control,	.21	.270	1.62	1.90	1.50	.90

The first four are "spent liquors," the last two columns are of "spent bark."

Taking all these figures together and comparing the results with the working tests as under Lœwenthal's and Hammel's methods,

it will be found that they are within 5% of the real value in all the figures, and within 3% in the first five columns, oak, hemlock, etc. From this it may be seen that the error in the execution of this method in ascertaining the percentage of tannic acid in an oak, hemlock, sumach, etc., would not exceed one ‡ of one per cent., while in the estimation of catechu, kino, etc., the error is quite large. The cause of this is that the tannate of copper precipitated from these last materials is contaminated with considerable other matter, and besides this the peculiar principles present in these materials exert a solvent action upon the tannate of copper. This method may be used for these last determinations, but the kino may be more accurately determined by Lœwenthal's method.

This method has been in use in my laboratory ever since I first investigated it, and the numerous analyses I am called upon to make of tanning materials and executed by it give the utmost satisfaction. I will continue to use it in all my leather researches now about to be commenced, also in commercial work. In conclusion I wish to tender my thanks to Mr. Jackson S. Schultz for valuable advice, and to all others who favored me with materials for this research, and facilitated it.

REVIEW OF INDUSTRIAL CHEMISTRY.

By. A. A. Brenneman.

Sulphuric Acid.*—*Lunge* has undertaken to study againt he phenomena of the lead chamber in order to test the conclusions put forward by R. Weber (†) in 1867 as to the action of SO_2 on NO and N_2O_5. He finds that SO_2 and NO do not react upon one another, even at 100° C. when both are dry, but in presence of water they act slowly, the reaction requiring 48 hours for its completion at 15°. N_2O is formed, but the reduction does not extend further, no N being detected. Sulphuric acid of 1.45 sp-gr. does not hasten the reaction unless free O is also present. N_2O is produced in the latter case even when O is in excess of the amount required by the reaction $2\,SO_2+2\,NO+3\,O+3\,H_2O=2\,H_2\,SO_4+2\,HNO_2$.

In the normal condition of the chamber both steam and O are in excess and a loss of nitrogen corresponding to the nitrate used in

(*) Ding. Jour. 243.1; Ber. D. Ch. Ges 14—2196. Chem. C. Bl. Dec. 7, '81.
(†) Ding. Jour. 184-246.

the pyrites burned must result, as N_2O is not absorbed by strong sulphuric acid in the Gay-Lussac tower.

The author also describes a new form of gas burette used in the investigation. *Laske* and *Benker* (*) propose to reduce the waste of nitric acid in the chamber process by two-thirds through the introduction of SO_2 at the outlet of the last chamber. NO_2 being reduced by this means to N_2O_3 which is more soluble in H_2SO_4, complete absorption occurs in the Gay-Lussac tower.

Richter (†) effects thorough mixture of the gases of the lead chamber by aspirating them through one opening in the wall of the chamber and injecting them again at another. A steam jet is used for the purpose.

SODA.—*Scheurer-Kestner*(‡) discusses the method of *Pauli* (§) for the oxidation of soda lyes. The oxidation of sulphites to hyposulphites and sulphates through the action of MnO_2 is considerable but still incomplete at a temperature of 75°.

Rammelsberg (‖) finds in the soda liquors from the manufactory of Schoenebecker, peculiar red and yellow crystals containing vanadium and yielding, on re-crystallization, colorless octahedra of the general formula, $NaF\ 2\,Na_3\ RO_4\ 18\,H_2O$, in which R may be P or V. He ascribes the origin of the phosphorus to the limestone, the ash of coal and to the clay of the furnace, the latter being also the source of the vanadium.

The same author(¶) finds crystals of Gay-Lussite ($Na_2CO_3\ CaCO_3\ 5\,H_2O$) in soda liquors from the above works and ascribes to the formation of this substance one of the principal losses of soda in the Leblanc process.

Reidemeister(††) finds the same substance to separate from crude soda lyes during carbonation, but never from the fully carbonated liquor. It also occurs in the lime mud of the causticising vats in the manufacture of caustic soda. It is insoluble in water, but is decomposed by prolonged action of cold water, more readily with the aid of heat. It forms most abundantly in concentrated caustic liquors. An anhydrous soda-lime carbonate occurring in over-

(*) Comptes Rendus, 92—181. Ding. Jour. 243.1.
(†) Ding. Jour. 243.1.
(‡) Ding Jour. 235-399.
(§) Comptes Rendus, 92.878. Ding. Jour. 242.4.
(‖) Ding. Jour. 242.4.
(¶) " " "
(††) Ding. Jour., 242.-4.

heated or "burnt" balls in the black ash process gives crystalline structure to the mass and hinders subsequent lixiviation. The temperature of the black ash furnace should therefore be maintained at a point below the melting point of this double carbonate.

W. Smith and *T. Liddle* (*) have shown that the loss of soda in the residue left from the lixiviation of black ash may be lessened, and the soda recovered by prolonged digestion with cold water, which decomposes the insoluble Gay-Lussite. "Soda waste" heated one hour at 60° yielded .22 per cent., Na_2O out of 2.4 per cent. of soda present, and a sample of causticising mud yielded by the same treatment 2.62 per cent. of soda, while 2.5—3 per cent. remained insoluble. By using this mud to replace lime in the balling process the insoluble soda is recovered, but when the mud is rejected a loss as high as 7 per cent. of soda may occur. Wright has already shown that the total loss of Na_2O in the Leblanc process may amount to 20 per cent. The writers confirm the statement of Scheurer-Kestner that an excess of lime in the balling furnace favors the production of insoluble compounds of soda during lixiviation.

The manufacture of soda by the ammonia process of Solvay has been improved by *Honigman*(†), who employs a moderately elevated temperature in the absorbing vessel, securing by this means a granular precipitate of bicarbonate instead of the slimy mud commonly produced in the cold. The precipitate is easily washed. After washing, it is pressed in the cakes which are decarbonated in an upright furnace, on the grate of which the cakes are exposed to a current of hot CO_2 from the generator. The dry powder falls through the grate, and the additional volume of CO_2 obtained is carried to the absorber.

Improvements in the apparatus of the ammonia process have also been made by *Montblanc and Gaulard* (‡), *Pechiney* (§) and by the *Societe Anonyme de Produits Chimique de Paris* (‖). *Ortlieb* and *Muller* (¶) apply the method of Solvay to the production of potassium carbonate, also through the substitution of trimethylamine for ammonia.

Trimethylamine, through the recent discovery of Camille Vincent, has become an abundant commercial product, being made

* Chem. News, No. 43. Ding. Jour., 242.4.
(†) Ding. Jour., 243.–1.
(‡) Ding. Jour. 243.1. (§) Ib. (‖) Ib.
(¶) Bul. Soc. Chimique, Jan. 5th, '82.

from the residues of beet root molasses. A single firm, Tilloy & Dulanne, of Coumeres, turn out 1800 kilos of crude salts of trimethylamine per day.

A current of CO_2 is passed into a solution of equal equivalents of potassium chloride and neutral trimethylamine hydrochloride at 23° C. and potassium carbonate ($KHCO_3$) is precipitated. The reaction is limited by the inverse reaction, but this difficulty is overcome by operating under pressure. An excess of trimethylamine (2.22 molecules to 2 KCl) has been found to increase the yield.

Lunge (*) has directed the examination of several reactions which have been suggested as indicating new methods of making soda, or of improving existing methods. The results are given as follows:

1. The action of Al_2O_3 on $NaNO_3$ at high temperatures with recovery of the nitric acid causes rapid attack of the vessels (platinum, iron and glass used) and HNO_3 is only partly recovered. The reaction is less available for manufacturing purposes than the earlier one of Schiappi, in which $CaCO_3$ and $NaNO_3$ are used.

2. The decomposition of sodium sulphate by a solution of calcium bicarbonate (*Pongowski's method*) (†) was found impossible using a three-fold quantity of pulverized marble, acting for ten hours upon sodium sulphate in a solution saturated with CO_2.

3. Aarland's process for substituting $CaCl_2$ for $MgCl_2$ in treatment of soda waste by the well known method of Schaffner and Helbig failed entirely when tried in the laboratory. $CaCl_2$ decomposes Ca S only after prolonged boiling, a result which may be obtained with water alone.

POTASH.—*Gruneberg* (‡) reviews the progress of the Stassfurt industry in its relation to the production of potassium sulphate from the complex salts and mixtures of the natural deposit. He discusses a list of 20 patents which have appeared since 1877, and classifies them as follows:—

1. Preparation of Schoenite ($MgSO_4$, $K_2SO_4 6H_2O$) from Kieserite ($MgSO_4$) and KCl or Carnallite ($K\ Mg\ Cl_3\ 6H_2\ O$)
2. Preparation of Schoenite from Kainite.
3. Working of mother liquors for by-products.
4. Production of K_2SO_4 from Schoenite by methods other than those depending on decomposition of KCl.

The author concludes that a combination of different processes

(*) Ding. Jour. 243 Heft. 2.
(†) French Patent, Mar. 27, 1872.
(‡) Ding. Jour. 243 Heft. 1.

will be found best in practice. In all processes it is advisable to use a minimum of water, and to have all waste liquors as rich in $MgCl_2$, and as poor in potassium salts as possible before they are rejected.

All methods of preparing potassium sulphate from these salts must compete with the direct method of the Leblanc process, by which the alkaline chloride is converted into sulphate, and are at an additional disadvantage in comparison with it, because of the loss of HCl in the waste liquors.

ABSTRACTS.

Abstracts from the *Comptes Rendus*, by A. Bourgougnon.

Complex Function of Morphine, and its Transformation into Picric Acid; also its Solubility. By M. Chastaing. No. 1. (Jan. 2nd, 1882).—Action of alkalies. Potassa, soda, baryta and lime dissolve morphia in the proportion of one equivalent of morphia for one equivalent of base. The solutions evaporated in a vacuum give crystallized products containing one equivalent of base, one equivalent of morphia and two equivalents of water.

Action of Nitric acid. Tetrahydrated nitric acid at a temperature of 100° C. transforms morphia into an acid with the formula $C_{20}H_{18}N_2O_{18}$, a tetrabasic acid crystallizing with difficulty. Baryta and lead salts of this acid contained $8\,H_2O$.

Monohydrated nitric acid, in sealed tubes at 100° C. transform the acid $C_{20}H_{18}N_2O_{18}$ into picric acid.

Solubility of morphia. 1 litre of water at 0° C. dissolve traces of morphia, at 10°, 0 gr. 10; at 20° 0 gr. 20; at 40° 0 gr. 40, of crystallized morphia. The solubility of morphia at 40° is then represented by a straight line, but above 45° the solubility is represented by a larger number than the elevation of temperature. From 45° to 100° the curve of solubility affects a parabolic form.

Carbonic Ether of Borneol. By M. A. Haller. No. 2. Jan. 9th, 1882.—This ether was found in the residues of preparation of the compound described by the author under the name of *Borneol Cyane*. When pure it affects the form of very light white needles or hexagonal tables, insoluble in water and the alkalies, not very soluble in cold alcohol, soluble in boiling alcohol, ether, chloroform, benzine, glacial acetic acid. Melting point 215° C. and sublimes without decomposition. Melted with caustic potash it gives potassium carbonate and borneol.

In the cold, nitric acid is without action, at 100° C. a combination takes place with the formation of an oily substance, if the temperature is raised above 100° there is a decomposition with formation of nitrous vapors and production of camphor. Formula, $C_{21}H_{34}O_3$.

Essential Oil of Savory. By M. A. Haller. No. 3. (Jan. 16, 1882.—This essential oil is obtained from Savory, (Satureia Montana L), it is a mixture of hydrocarbons and phenols, and does not contain camphor; it is a liquid of an orange color, not very fluid, of an aromatic odor. Sp. gr. 0.7394 at 17° C. Rotatory power for a stratum of 200 m.m. is LD= −6°5 at 17° C. Treated withcaustic soda an insoluble hydrocarbon is produced; if after the separation of the insoluble hydrocarbon, the alkaline solution is acidified, an oily liquid is produced which distils between 230° and 245° C. After several rectifications a liquid is obtained boiling at 232°—233° C. Sp. gr. 0.972 at 17°, soluble in alcohol, ether and alkalies. It has for formula $C_{10}H_{14}O$ and is isomeric with thymol.

Diatomic Alcohol Derived from Beta-Naphthol. By G. Rousseau. No. 3. (Jan. 16, 1882.)—Beta-naphthol treated with chloroform and caustic soda in solution gives, besides an aldelyde, a white product characterized as a pseudo-diatomic alcohol.

It melts at 230°C. with decomposition, nearly insoluble in benzine, carbon-disulphide and acetic acid, it is dissolved in small quantity in ether and petroleum naphtha.

An analysis has given the following numbers.

	I.	II.
C	86.01	85.94
H	4.65	4.60

The calculation for $C_{22}H_{14}O_2$ gives

C	85.16
H	4.51

Phosphoric Acid in the soils of the North of France. By A. Ladureau. No. 3. (Jan. 16, 1882.) **Silicomolybdic Acid.** By F. Parmentier. No. 5. (Jan. 30th, 1882.)—Obtained by the action of hydrochloric acid upon silicomolybdate of suboxide of mercury, the liberated acid gives by evaporation large bright-yellow cubo-octahedral crystals melting at about 45°, very soluble in water and diluted acids, forming crystallized salts with bases. Its composition is represented by SiO_2 12 MoO_3 + 26 H_2O, corresponding to the silicotungstic acid of Marignac.

Specific Gravity of Vapor of Pyrosulfuryl Chloride. By J. Ogier. No. 5. (Jan. 30th, 1882.)—Victor Meyer's and Dumas' method have been employed.

The first method gives the following numbers: 3.88, 3.59, 3.99, 3.69, 3.36; mean 3.70 in anilin vapor. The results obtained in sulphur or mercury vapor do not differ very much; they are 3.72, 3.42, 3.30. By Dumas' method the numbers obtained are 3.87, 3.72, 3.69, 3.70; average 3.74 at ordinary pressure, and at temperatures between 160° and 200°C.

The pressure being reduced (200 to 300^{mm} of mercury) in order to obtain a decrease of temperature, the sp. gr. obtained at 170° were 3.69, 3.75, 3.80, 3.69, average 3.73. The vapor weighed was ascertained by analysis to be a pure product.

Researches on Pilocarpin. By M. Chastaing. No. 5. (Jan. 30th, 1882.)—Pure pilocarpin has been treated with melted potassa, a volatile product with an alkaline reaction was formed, giving a precipitate with a slightly acid solution of platinum chloride, forming methylamin chloroplatinate. There has been no formation of a volatile base having the characteristics of conicin. Under the influence of melted potassa, in excess, pilocarpin furnishes methylamin, carbonic acid, butyric acid, and traces of acetic acid.

FOREIGN PATENTS.

C. Arnold, New York: *Apparatus for generating ozone* (Germ. P. 15678, Jan. 20, 1881). Into a tray of glazed earthenware are placed vessels containing pieces of phosphorus and water. The tray is covered with a bell of unglazed, porous earthenware. Through this bell the air passes in and the ozone passes out whilst the oxidation products of the phosphorus remain inside and dissolve in the water contained in the glazed tray.

H. Precht, New Stassfurt: *Method of preparing magnesia, potassium sulphide and potassium sulphate from potassium-magnesium sulphate.* (Germ. P. 15,747, March 8, 1881). The manufacture of potassium sulphate from the above mentioned double sulphate by heating it to redness by carbon, according to the equation:—
$2\,(K_2SO_4\,MgSO_4) + C = 2\,K_2SO_4 + 2\,MgO + 2\,SO_2 + CO_2$;
as indicated by Schwarz (Dingler's polyt. J. vol. 219, p. 352), is

modified so as to first produce potassium sulphide in the mixture. This sulphide effects the decomposition of the double sulphate with greater energy, rapidity and completeness.

I. EGESTORFF, Hanover: *Improvements in salt evaporating pans and method of filtering brine.* (Germ. P. 14,782, Feb. 1, 1881). Mechanical arrangement for protecting the steam coils from the depositing salt, and filtration of the hot brine through flannel before running it into the pan.

A. R. PECHINEY, Salindres: *Method of preparing chlorates.* (Germ. P. 15,493, March 10, 1881). By the usual method of manufacturing potassium chlorate, decomposition of the product resulting from the action of chlorine upon milk of lime, by means of potassium chloride, a part of the potassium chloride remains dissolved in the calcium chloride mother liquor, after the crystallization of the potassium salt. In order to reduce this loss to a minimum the inventor removes the greater part of the calcium chloride from the mixture of calcium chlorate and chloride before adding the potassium chloride. This he effects by evaporating the liquid to 48°Be. and cooling, whereupon calcium chloride crystallizes, and can be removed, or by adding 3 molecules of lime for 1 molecule of calcium chloride whereby basic chlorides are formed, which can be separated from the liquid, or finally both operations may be employed consecutively.

C. LUCKOW, Deutz: *Electrolysis of zinc solutions for the purpose of extracting zinc from its ores.* (Germ. P. 14,256, April 20, 1880). Concentrated solutions of zinc salts are electrolized. The zinc is separated in grains, and the acids liberated are made to act upon crude or wasted zinc ores connected with the anode of the battery. Polarization of the electrodes is neutralized by chemical or mechanical means.

A. GURLT, Bonn: *Method of working up calamine residues and zinc waste by treatment with gaseous or liquid hydrochloric acid in combination with chlorides of the alkalies or alkaline earths.* (Germ. P. 15,012, Dec. 5, 1880, 1st additional patent to No. 8,116). The modification of the original patent consists in the employment of gaseous or liquid hydrochloric acid during the distillation of the zinc chloride, whereby the extraction of the zinc is facilitated.

P. MANHES, Lyons: *Manufacture of refined copper from copper mattes in Bessemer converters by the use of reducing agents.*

(Germ. P. 15,562, Aug. 20, 1880). Sulphurous copper ores are melted down in a pit furnace. The matte obtained is treated with air in a converter in the same manner as pig iron in the Bessemer process. The operation is ended when evolution of sulphurous acid has nearly ceased. The product is crude copper. To refine it, charcoal powder is blown in with the air. Modifications of the process are described for the treatment of mattes rich in iron or too deficient in sulphur to produce the required degree of fluidity.

MAX TSCHIRNER, San Francisco: *Improvements in the manufacture of explosive compounds.* (Germ. P. 15,508, Feb. 6, 1880). Picric acid, 1 part; tar, 1 part; and potassium chlorate, 5 parts.

I. M. LEWIN, Paris: *Use of cotton and dextrine for the preparation of a gelatinous nitro-glycerine.* (Germ. P. 15,073, Jan. 18 1881). Purified and finely divided cotton is mixed with 5 parts of dextrine and heated under pressure in a closed vessel. The jelly thereby formed is dissolved in nitro-glycerine to the extent of 7 per cent., and forms a mass from which the nitro-glycerine will not drain. An explosive compound is formed by mixing this gelatinous nitro-glycerine with nitre and sawdust.

CHEMISCHE FABRIK AUF ACTIEN, Berlin: *Preparation of coloring matters from sulphosalicylic acid.* (Germ. P. 15,117, Dec. 10, 1880, and Germ. P. 15,889, Mar. 18, 1881). Sulphosalicylic acid is nitrated by means of nitric acid of sp. gr. 1.35. Stronger nitric acid eliminates the sulpho group with formation of various nitro-compounds. Nitro-sulphosalicylic acid and its soluble salts dye silk and wool yellow. The coloring power is augmented by the introduction of one atom of bromine. Excess of bromine also eliminates the sulpho group, forming dibromnitrosalicylic acid.

Sulphosalicylic acid also reacts with phenols to form coloring matters, yielding, for instance, with resorcin a brownish-red dye.

With diazo, diazoamido, and amidoazo compounds, sulphosalicylic acid produces red dyes.

In Patent No. 15,889, the method of preparation of the bromine substitution compounds of introsulphosalicylic and nitrosalicylic acids is claimed.

UPON THE PREPARATION OF ARTIFICIAL INDIGO AND SOME OF ITS DERIVATIVES.

By James H. Stebbins, Jr., S. B.*

It will be needless for me to tell you what indigo is, as you have probably all come across it, and know whence it is derived.

I will therefore only give you a graphic description of how this interesting body can be made synthetically; and in giving you this description, I can do no better than quote Baeyer's wonderful experiments :

Baeyer says : "The first thing I attempted to do was to remove the oxygen in isatine, and it will be remembered how this was accomplished, in 1865 and 1866, by the conversion of isatine into dioxindole, oxindole, and indole."

It was then tried to find an easier method for the production of indole, as Baeyer considered it to be the basis of the indigo group.

In 1868 Baeyer obtained it directly, by treating indigo with zinc dust ; and in 1869 he produced it synthetically from nitrocinnamic acid and its reduction product.

The following quotation may help to explain Baeyer's reasoning more thoroughly :

"If it is desired to make indole synthetically, it becomes necessary to introduce into benzol a diatomic carbon chain and a nitrogen atom, and then to connect the one with the other. The properties hereby necessitated will be found realized in nitrocinnamic acid, which has been deprived of carbonic acid, and the oxygen of the nitro group ; and, in fact, as we have already found, nitrocinnamic acid on being fused with potash produces indole."

Shortly after this, Kekule informed us that he had produced in a similar way isatine, from orthonitrophenylacetic acid. Baeyer and Suida then examined this compound more closely, and came to the conclusion that dioxindole and oxindole could be nothing else than the anhydrides of orthoamido-amygdalic acid and orthoamido-phenyl acetic acid. A synthetical examination soon after followed, and showed that oxindole by oxidation is converted into isatine ; which, however, was shortly afterwards produced by Claisen and Shadwell, by a new and convenient synthetical method.

In 1870, Baeyer and Emmerlin converted this isatine into indigo, and thus solved the problem he had so long been working at ; but, as the materials used (phosphorous trichloride, phosphorous, and acetyl chloride) were quite complicated, he strove to find a more simple method for accomplishing this end, and finally came to the

*Read at the January Conversazione.

conclusion that it would be better to first treat the isatine with phosphorous pentachloride, and then phosphorus, or some other reducing agent.

It was then tried to produce, in the same way, indole from oxindole; and, in fact, a body answering to the formula of a dichlorindole was obtained.

Without taking up any more of your time with the history of Baeyer's discoveries, I will now describe some of the products which may be converted into indigo blue.

ORTHONITROCINNAMAC ACID.

This acid may be easily prepared, according to Beilstein and Kullberg's process, and likewise the separation of the ortho from the paranitro compound by means of ether.

This acid bears a simple relation to indigo, as may be seen from the following formulæ:

Nitrocinnamic Acid. *Indigo.*

$$C_9\,H_7\,NO_4 = C_8\,H_5\,NO + CO_2 + H_2O$$

ORTHONITRODIBROMCINNAMIC ACID.

This acid is produced by treating the nitro compound with bromine under exclusion of sunlight.

The bromine compound crystallizes in short colorless needles, which melt at 180° C., under decomposition and formation of a small amount of indigo.

This body was found to have the formula $C_9\,H_7\,(Br_2)\,NO_4$ = orthonitrodibromcinnamic acid.

This body is soluble in caustic alkalies, without decomposition; which, however, by heat is converted into orthonitropropiolic acid, and then into isatine. By heating an aqueous solution of the above acid with carbonate of soda, a small amount of crystallized indigo blue is the result.

ORTHONITROPHENYLPROPIOLIC ACID.

$$C_9\,H_5\,NO_4$$

The bearing of orthonitrodibromcinnamic acid with alkalies differs considerably from the non-nitrated compound, inasmuch as HBr may readily be removed from the former, whereas from the latter it can only be removed by the energetic action of alcoholic potash to form propiolic acid. The propiolic acid is prepared usually from an aqueous solution of the dibrominated compound by treating with an excess of caustic soda solution.

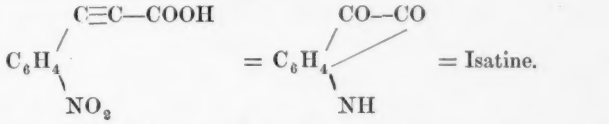
The compound is then thrown down by means of an acid, and dried. The orthonitrophenylpropiolic acid so obtained appears as little colorless needles, melting at 155—160° C. with sudden decomposition.

ORTHONITROPHENYLACETYLEN.

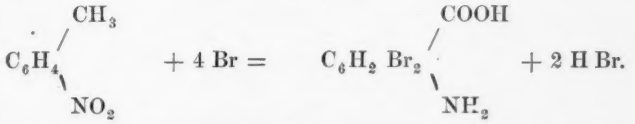
This compound may be obtained from orthonitrophenylpropiolic acid, by boiling with water in an ascending cooler. It crystallizes from alcohol, in colorless needles, and melts at 81–82° C.

FORMATION OF ISATINE FROM ORTHONITROPHENYLPROPIOLIC ACID.

Isatine may be obtained in abundance by boiling a solution of orthonitrophenylpropiolic acid with alkalies. The reaction that takes place here may be explained as follows :



In other words, CO_2 is split off; at the same time a reduction of the NO_2 group, and oxidation of the side chain takes place. This reaction is similar to the reaction which takes place when bromine is allowed to act on orthonitrotoluol, viz :



FORMATION OF INDIGO FROM ORTHONITROPHENYLPROPIOLIC ACID.

If a very dilute alkaline solution of the acid be heated to boiling, and a small piece of grape sugar is added thereto, the liquid first turns blue, and then follows a copious precipitation of small blue needles, with coppery red lustre. These needles are perfectly pure indigo-blue. The yield so obtained is about 40% of the propiolic acid used.

The formation of indigo-blue in this case takes place as follows :

First the alkali, acting on propiolic acid, splits off CO_2, forming isatine, which, however, on being treated with a reducing agent as grape sugar, loses an atom of oxygen and forms indigo-blue, viz :

$$C_9H_5(NO_2)\,O_2 = \underset{\text{(Indigo.)}}{C_8H_5NO_2} + CO_2$$

$$C_9H_5(NO_2)\,O_2 = C_8H_5NO + O + CO_2$$

ORTHONITROPHENYLCHLORLACTIC ACID.

This acid may be easily prepared by passing a current of chlorine into an alkaline solution of orthonitrocinnamic acid. The mixture so produced is composed of orthonitrochlorstyrol, and orthonitrophenylchlorlactic acid, from which the latter may be separated by acidifying the mixture and then extracting the orthonitrophenylchlorlactic acid with ether. This compound appears as a white crystalline mass, and having a melting point of 119°—120° C.

Its formula may be represented by

$$C_6H_4\begin{cases} NO_2 \\ C_2HCl.\,COOH. \end{cases}$$

This compound on being treated with reducing agents, such as sodium amalgam, or sulphate of iron in an alkaline solution, forms indole.

ORTHONITROPHENYLOXACRYLIC ACID.

$$C_6H_4\begin{cases} NO_2 \\ C_2HOH.\,COOH. \end{cases}$$

This compound may easily be produced from orthonitrophenylchlorlactic acid, by treating with alcoholic potash. It crystallizes from hot water, in flat needles.

If heated slowly to 110° C. it melts, giving off CO_2, and forming a dark blue mass, which, on being treated with alcohol, dissolves with a brown color, in which small crystals of indigo-blue remain suspended.

Indigo-blue may also be derived from the above by heating its solution with phenol, but the yield of coloring matter so obtained is quite small.

The reaction taking place here may best be explained as follows:

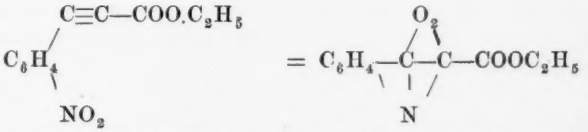
$$C_9H_7NO_5 = C_8H_5NO + CO_2 + H_2O + O.$$

The ease with which orthonitrophenylpropiolic acid is converted into indigo-blue, with such mild re-agents as grape sugar, appeared very strange to Baeyer, and in the following I will detail the experiments he made to arrive at a clear understanding of the above mentioned re-action.

By treating orthonitrophenylpropiolic acid with strong H_2SO_4, at

the ordinary temperature, its isomer isatogenic acid ether is obtained, which crystallizes in yellow needles and melts at 115° C.

The formation of this compound may be explained by the following re-action :



A solution of nitrophenylpropiolic acid in H_2SO_4, on being treated with reducing agents, forms a new blue dye-stuff, called indoine. Indoine is very similar to indigo blue but differs from it in that it dissolves in strong H_2SO_4 with a blue color, which, on being heated, only forms a sulpho acid with great difficulty.

INDOXYLIC ACID ETHER.

This body is produced by the action of reducing agents, on isatogenic acid ether. It appears in the shape of thick prisms, and melts at 120—121°C.

It has the formula $C_{10}H_{11}NO_3$.

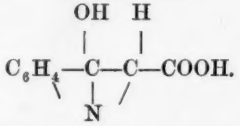
This compound on being rapidly heated forms a little indigo-blue; but if heated to 100°C. with strong H_2SO_4 it passes over into indigo sulpho acid almost quantitatively.

On being fused with caustic soda, at a temperature of 180°, it forms indoxylic acid, which melts at 160°C.

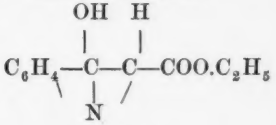
Ethyl indoxylic acid and nitrotrosoethylindoxylic acid have likewise been produced, but, I will not attempt to describe their formation.

The following formulæ will best represent the compounds just enumerated.

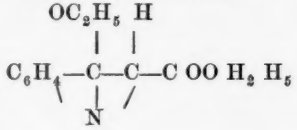
Indoxylic acid.



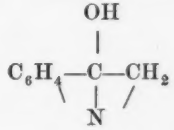
Indoxylic acid ether.



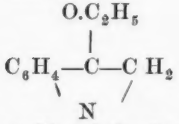
Ethylinolxylic acid ether.



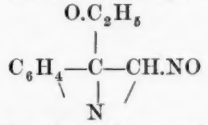
Indoxyl.



Ethyl indoxyl.



Nitrosoethyl indoxyl.



Indoxylic acid on being heated as high as its melting point with H_2SO_4, gives off CO_2, and passes over into indoxyl.

If indoxyl be dissolved in dilute KOH. and exposed to the air, indigo-blue is formed, which falls out as a blue precipitate.

A mixture of indoxyl and orthonitrophenylpropiolic acid, on being treated with H_2SO_4 forms indoine.

Indoxyl and nitrophenylpropiolic acid on being treated with Na_2CO_3 are immediately converted into indigo-blue.

A mixture of indoxyl and isatine in alcoholic solution, on being treated with Na_2CO_3 forms indirubine, which appears as brownish-red needles, with metallic lustre.

Thus far we have only considered the formation of indigo-blue from a theoretical point of view, and I will now try to describe the way it is made upon a large scale, or, rather, the manner in which it may be produced, and fixed in, or upon the fibre of materials at the same time.

The formation of indigo-blue within the fibre of materials to be printed, depends upon the facility with which orthonitrophenyl propiolic acid is converted into indigo-blue, by the action of reducing agents.

The reducing agents more commonly used are grape sugar, and especially the xanthates of soda or potash.

In other words, to print a pretty blue upon calico, the orthonitrophenylpropiolic acid is mixed up with a starch thickening, and xanthate of soda, and borax, and then printed in the usual way upon the calico. This is allowed to dry, and then heated to 100°C. for a certain length of time, which process develops the color within the fibre.

PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

Adjourned meeting held Friday, May 19th, 1882.

The meeting was called to order at 8:40 P. M., Dr. Chas. A. Doremus in the chair.

The first paper of the evening, "On the Examination of Some of the Methods for the Determination of Phosphorus in Iron Ores," by Dr. E. Waller was then read.

After giving the results obtained by methods suggested or recommended by Prof. Classen and M. Derome, Dr. Waller stated that he found a fusion of the pulverized ore with 2 to 3 times its weight of acid sulphate, finally using the blast lamp, afforded the phosphoric acid in the ore in a form soluble in dilute nitric acid, from which it could be separated by molybdate solution.

Dr. Prochazka spoke of the advantages of using Finkener's process (*Berichte Deut. Chem. Ges.* xi. 1638), in which most mineral acids can be used as solvents; and further stated that in applying this process, hydrochloric acid did not interfere with the precipitation of phosphoric acid. To get a complete and rapid precipitation, the solution should contain at least 20 per cent. of ammonium nitrate.

After some questions by Mr. Pitkin, the second paper, "Report of Progress in Industrial Chemistry," by A. A. Brenneman, was read by the Recording Secretary.

Prof. Brenneman described improvements in the manufacture of carbonate of soda, alum, &c., giving an interesting account of each.

After some remarks, Dr. Waller exhibited a piece of apparatus designed by Wm. Bettel, of England, for the estimation of nitrogen in water, beer, &c. The important feature of the apparatus consisted in a peculiarly constructed copper flask, in which by a continuous operation the ammonia is distilled off, and (indeed) a combustion of the residue with caustic soda is effected. It is claimed by the inventor to give good results.

After which the meeting adjourned.

JAMES H. STEBBINS, JR.,
Recording Secretary.

XV. AN EXAMINATION OF SOME OF THE METHODS FOR DETERMINING PHOSPHORUS IN IRON ORES.

By E. Waller, Ph. D.

The present investigation was undertaken with a view to obtain a more rapid method of determining phosphorus than those at pres- in use, by utilizing some suggestions which have recently been made.

Two lines of experiment were followed: That sugested by Prof. Classen (*Quantitative Analyse auf Electrolytischem Wege, Aachen* 1882), and that of M. P. Derome (*Comptes Rendus, LXXIX*, 925 translated in *Chem. News*, XL, 292).

For the purpose two limonites were taken, which by ordinary methods of analysis yielded as follows:

	"High"	"Low"
Fe	54.50	52.35
P	1.06	0.08

These had been pulverized and passed through an "80 mesh" sieve.

Electrolytical Separation (Classen).

In this the idea was to separate the iron by the use of the battery, thus incidentally determining the percentage of iron, and then, in the solution thus freed from the disturbing influence of such large quantities of iron (relative to the amount of phosphorus), to determine the phosphorus.

An experiment with an indefinite amount of ferric chloride showed that it took about two hours to deposit a little over 0.5 gm. of iron by the battery. The conditions, as specified by Prof. Classen, are that the solution—nearly neutralized—must receive the addition of a large excess of ammonium or potassium oxalate, and that the electrolysis shall be carried on in the solution heated to about 80° C. Three freshly charged Bunsen cells, of about 3 pints capacity each, were used. Prof. Classen states that enough oxalate must be used to form the double oxalate (of iron and ammonia or potassa) and then 3 or 4 gms. more of the oxalate must be added. The action of the battery converts the oxalate into bi-carbonate, and any deficiency of oxalate is soon perceived from the appearance of flocculent masses of ferric hydrate in the solution. It was found that considerably more oxalate in excess had to be used than the amount specified by Dr. Classen.

As so much oxalate was required it was not deemed possible—even with a platinum dish of over 250 c.c. capacity—to use more than 1 gm. of ore for the determination. The ore was fused with alkaline carbonates, dissolved in dilute HCl, and evaporated down to separate silica, taken up with dilute HCl, filtered, nearly neutralized, and the oxalate added. In the first experiment on the "High" ore only ammonium oxalate was used, and in this case, after a certain time, a flocculent precipitate of ferric hydrate formed, which refused to dissolve on addition of more alkaline oxalate, or to reduce to metallic iron, though the battery was allowed to act upon it all night. The solution, with precipitate, was therefore poured off and examined for phosphoric acid.

The substitution of potassium oxalate in part for ammonium oxalate, a proceeding suggested, though not insisted upon, by Prof. Classen, remedied this difficulty, though the amount of oxalate necessary was so great that the solution, when cold, was nearly solid with the interlocking crystals of the alkaline salts.

The action of the battery, aided by the temperature necessary, (80° to 100° C.) kept the solution in such a lively state of effervesence that some of the solution was inevitably lost mechanically. When the solutions gave no reaction for iron they were poured off, the dishes rinsed with hot water, and finally with alcohol and ether. The deposited iron was not quite smooth, apparently containing granules of what was probably alumina hydrate or phosphate, which tenaciously adhered, and in some cases appeared to be partially covered with a coating of metallic iron. The solutions also were slightly turbid, apparently from the presence of alumina compounds. These solutions were evaporated down twice with concentrated nitric acid, then diluted and filtered into a molybdate solution containing 10 or more gms. of ammonium nitrate; the molybdate precipitate, after heating for some hours, filtered off, washed, dissolved in ammonia and the phosphoric acid separated by magnesia mixture. The results were :

	"High"	"Low"
Fe	56.01	50.45
P (Fe partly separated)	1.04	
P (Fe entirely separated)	0.988	0.089

Prof. Classen remarks (p. 29): "In presence of alumina the electrolytic method gives but little advantage in the determination of phosphoric acid. In this case a precipitation of that constituent in a separate portion with molybdate solution is to be preferred."

The observation might obviously have been applied to the determination of iron also.

Sulphate Method (Derome).

M. Derome's method consists in fusing the mixture of ferric oxide, alumina and phosphoric acid, (precipitated by ammonia or basic acetate), with five to six times its weight of dry sodium sulphate over a blast lamp for 8 to 10 minutes, and then extracting with water. In the solution thus obtained it is asserted that the phosphoric acid can be directly precipitated with magnesia mixture. The ores were fused, &c., as above described, and the amount of filtrate from silica, representing 1 gm. of ore, precipitated with ammonia, washed, dried, ignited and fused as prescribed. To insure the change to sodium phosphate, the fusion was kept up for 15 minutes at the highest attainable heat of the blast lamp.

The water solution was found to contain small amounts of iron and manganese, possibly some other compounds, so that the addition of magnesia mixture gave a precipitate containing much beside pure magnesium ammonium phosphate, and it was found necessary to separate by molybdate solution before using the magnesia mixture. The precipitate, taken just as it was, after ignition, appeared to be too coarse to permit of the satisfactory action of the sulphate, so in one experiment it was pulverized finely before fusing. The results for phosphorus were :

	"High."	"Low."
Precipitate fused direct.....	0.669	0.028
" pulverized and fused.....		0.058

Experiments were also made as to the effect of fusing the ore direct with the neutral sulphate. On account of the presence of small amounts of lime (under 1 per cent.), the mass after fusing was extracted with acid. Hot acetic acid took out much of the phosphoric acid which refused to dissolve in water, but interfered perceptibly with the precipitation by molybdate solution. Dilute nitric acid was found to dissolve what would dissolve out satisfactorily, the greater part of the iron oxide remaining undissolved in crystalline scales. The results were :

	High.	Low.
Ore fused with $Na_2 SO_4$	0.356	0.0194

The use of some acid sulphate was then tried; 3 gms. of each ore were fused with 3 gms. Na HSO_4 at a low red heat until fumes ceased to come off ; then 3 gms. $Na_2 SO_4$ were added, and the whole

fused at the highest heat of the blast lamp for ten minutes. On cooling the mass was extracted with about 200 c.c. of water, to which was added 10 c. c. conc. nitric acid, and after heating for half an hour, at just short of boiling, the solution was filtered into molybdate solution into which some 10—15 gm. of crystals of ammonium nitrate had been thrown, &c., the phosphorus being finally weighed as the magnesium compound. (See result below). The neutral sulphate appeared to give but little advantage, and in this case the proportion of acid sulphate was evidently insufficient to completely disintegrate the ore. The experiment was therefore repeated, using three times as much $Na\,HSO_4$ as ore ; fusing for some time (one hour or a little over) at a low heat until fumes had almost ceased to come off, and then using the blast lamp for full ten minutes, extracting with dilute nitric acid, &c., as before. The results in the two last experiments were :

	High.	Low.
Ore fused with equal weights, $NaHSO_4$ and $Na_2\,SO_4$,	0.98	0.067
Ore fused with three times its weight, $NaH\,SO_4$,	1.05	0.083

This last mode of procedure, then, appears to be satisfactory for a rapid method of determining phosphorus in an iron ore. The amount of iron going into solution in the nitric acid is extremely small, so that that disturbing influence is removed. I propose making further experiments on the application of this method to titaniferous and other ores, and to bar iron and steel.

PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

Room 1 *University Building, N. Y., regular meeting June* 3*d*, 1882.

Dr. A. R. Leeds in the chair.

The minutes of the previous meeting were read, and after some corrections, were approved.

The minutes of the meeting of the Board of Directors on May 9, 1882, were then read.

The following gentlemen were then unanimously elected as regular members:

G. E. Bailey, proposed by M. Benjamin, W. Rupp and A. H. Elliott; Dr. Wm. Strattford, of the College of the city of New York, proposed by Chas. A. Doremus, Jas. H. Stebbins Jr., and A. R. Leeds; H. Edward Stockbridge, proposed by E. H. Goessmann, H. Endemann, and Jas. H. Stebbins Jr.; J. E. Mallett, Hoffman House, N. Y., proposed by C. A. Doremus, Jas. H. Stebbins Jr. and A. R. Leeds.

As associate member:

Y. Polledo, School of Mines, Columbia College, proposed by T. O'C. Sloane, Jas. H. Stebbins Jr. and A. H. Elliott.

The Librarian reported that Nos. 1 to 9 of the *Chemiker Zeitung*, and also some numbers of the *Berichte der Deutschen Chem. Ges.* were missing, and had been improperly removed from the library.

After which the following paper was read: On "Acetate of Lime, its manufacture and analysis," by Messrs. C. M. Stillwell and T. S. Gladding.

Dr. Waller remarked that the presence of acetic acid interferes decidedly with the precipitation of phosphoric acid by molybdate solution.

Mr. Stillwell replied that he overcame this difficulty by using large quantities of ammonium nitrate.

Dr. Grothe asked if the authors had noticed the presence of acetone in the products of the decomposition of acetate of lime by heat.

Mr. Stillwell said he had not noticed it particularly, since his attention had been confined simply to the loss of acetic acid.

Dr. Squibb remarked that it was not necessary to carbonize the wood, but that all the acetic acid could be obtained at a much

lower temperature, (about 160 to 200° C.) and the products then contain no acetone.

For the analysis of sodium acetate he uses a glass retort covered with copper gauze, and heats it on one side to prevent frothing and bumping. He uses 10 gms. of the acetate with 20 c.c. of water, and 10 c.c. sulphuric acid; after distilling off 10 to 15 c.c. of liquid, the distillation is interrupted,—about 20 c.c. of water are added to the retort, and 10–15 c.c. of liquid again distilled over. This is repeated a third time when all the acetic acid will have come over, without trouble from frothing or bumping during distillation.

Dr. Squibb further remarked that he used in his factory, retorts 20 feet long, 2 feet wide, and 10 feet deep, holding 2½ cords of wood, and heats them in a hot air bath. During the first twelve hours, only water is given off. In about 24 hours the acetic acid begins to distill over, the process being completed in 6 to 7 days for each retort.

Seasoned oak is preferred, and any admixture of softer woods diminishes the yield of acetic acid. Chestnut is notable in this respect.

Throughout the active heating, but more copiously toward the end, a gas, colorless and odorless (and, so far as tested, uninflammable), comes over, having anæsthetic properties. This gas adheres to the wood, after the charge is cooled and drawn, and seems to be a reason why small vermin will not remain near it. If the charge is heated too long, smoke appears at the exit pipe and carbonization of the wood begins at the centre of the top of the charge, extending in V-shape toward the bottom. When once started this carbonization proceeds spontaneously without further application of heat. Indeed it is sometimes found difficult to check it, even by the liberal application of cold water to the outside of the retort.

In the course of the operation, when properly managed the charge shrinks one third of its volume. 4000 lbs. of wood yield about 2800 lbs. of residue. The residue retains all the appearances of the wood before distillation, only that it becomes walnut-colored and it has the same elementary composition as that of kiln-dried wood. It is brittle and not well adapted to construction, but forms a most excellent fuel for many purposes, especially for kindling anthracite coal. The distillate is neutralized with soda ash and distilled to about one-fortieth its volume. The first product is crude wood spirit. This is redistilled, and gives rectified wood

spirit, and lastly wood oils, which contain large quantities of furfurol and no acetone. The wood oils are separated by passing the last portions of distillate into water. The rectified wood spirit contains about 80 % of methyl acetate, and when saponified gives a very pure methyl alcohol.

One cord of well-seasoned wood will afford 1200 to 1400 lbs. of liquid products. A cord of oak yields 60 to 70 lbs. of glacial acetic acid.

Dr. Squibb further remarked that there was no process commercially practicable for obtaining pure acetic acid from pyroligneous acid, or acetate of lime, but only from acetate of soda. Mr. Parker remarked that he had tried a process for making acetic acid by heating wood fibre with steam, under a pressure of 60 lbs. at 275° C. and confirmed Dr. Squibb's observations on the deterioration of the woody fibre, and also the impracticability of making pure acetic acid from pyroligneous acid.

Dr. Grothe remarked that in distilling acetone with bleaching powder, he had obtained chloroform simultaneously with calcium acetate.

Dr. Doremus brought up the question of giving reprints of papers to members. After some discussion Mr. Casamajor moved that "those members who desire reprints of their papers shall notify the committee on papers and publication, who may make arrangements to have them supplied at the expense of the authors." Carried.

After which the society adjourned to September 1st, 1882.

ARTHUR H. ELLIOTT,
Rec. Sec. pro tem.

XVI. ACETATE OF LIME—ITS MANUFACTURE AND ANALYSIS.

BY STILLWELL & GLADDING.

Acetic acid and its compounds hold a very important place in the arts and manufactures. They are extensively used in many branches of industry, as, calico-printing and dyeing, in the treatment of gums, in the manufacture of paints and varnishes, etc. Acetic acid is well known in the form of vinegar.

It may be formed in three ways: (*a*) by acetous fermentation, chiefly used for the making of vinegar; (*b*) by destructive distilla-

tion of wood, yielding crude pyroligneous acid and by further treatment, acetates of iron, lime, lead, soda, etc.; (*c*) by the distillation of various metallic acetates with sulphuric or hydrochloric acid, yielding the pure acid of commerce.

We will confine ourselves for the present to the second method of obtaining this acid—by the destructive distillation of wood:

"Wood consists essentially of woody fibre or cellulose, a small amount of mineral matter and sap and a variable amount of hygroscopic water. The woody fibre constitutes about 95 per cent. of dry wood, and is composed of, in 100 parts: carbon, 44.45; hydrogen, 6.17; oxygen, 49.38. The sap consists chiefly of water, holding organic and inorganic matters partly in solution and partly suspended. The inorganic constituents of the sap (the ash left after the incineration of the wood) are the same in all kinds of wood. In practice it is assumed that wood leaves about 1 per cent. of ash; but there is a difference for certain portions of the tree, the trunk yielding about 1.23 per cent. of ash, the branches and knotty parts 1.34 and 1.54, and the roots 2.27 parts of ash respectively.

Wood dried at 266° Fah.—at which temperature all the hygroscopic water is driven off—is composed of :

50 parts of carbon (inclusive of one part ash).
50 " chemically combined water."

In the process of dry distillation the hygroscopic moisture of the wood is first driven off. As the heat increases, acetic acid and water are formed by the decomposition of the wood, then follow the tar and volatile oils and at a still greater heat carbonic acid, carbonic oxide and marsh gas appear. The remainder of the carbon is left in the retort in the form of charcoal.

The following table from Wagner's Chemical Technology, shows at a glance the numerous and complex products which are formed during the destructive distillation of wood:

- Wood.
  - 1. Real wood.
  - 2. Hygroscopic water.
  - *a.* Illuminating gas. — Acetylen. Carbonic oxide. Elayl. Carbonic acid. Benzol. Marsh gas. Naphthalin. (?) Hydrogen.
  - *b.* Tar. — Benzol. Oxyphenic acid. Naphthalin. (?) Cresylic acid. Paraffin. Phlorylic acid. Reten. Empyreumatic resins. Carbolic acid. Creosote.
  - *c.* Pyroligneous acid — Acetic acid. Aceton. Propionic acid. Wood spirit.
  - *d.* Wood charcoal.

Woods differ greatly in the amount of water which is present in them. The following table by Schuebler and Hartig shows the amount of water present in some freshly cut woods:

Hornbeam	contains	18.6	per	cent.	of water.
Willow,	"	26.0	"	"	"
Ash,	"	28.7	"	"	"
Birch,	"	30.8	"	"	"
Oak,	"	34.7	"	"	"
Pine,	"	39.7	"	"	"
Red Beech,	"	39.7	"	"	"
Elm,	"	44.5	"	"	"
Larch,	"	48.6	"	"	"
White Poplar,	"	50.6	"	"	"

Different woods yield different results, hence the wood selected must be chosen with reference to the nature of the products desired. If the charcoal is to be used for making powder, alder, willow and dogwood are used; if for heating, oak and beech; if for pyroligneous acid alone, birch, thorn, and apple. Oak holds the first place and beech the second when we take into account all the products obtained—charcoal, acetic acid, naphtha, and tar.

The following table, by Stolze, shows the various products obtained from different woods:

336 lbs. wood.	Charcoal.	Acid Liquor.	Sp. Gr.	Dry Acetate of Lime.
Beech	84	180	1.029	25
Walnut	72	150	1.018	14½
Birch, cut 3 years	70	120	1.031	13
Oak	91	190	1.022	24
Ash	90	190	1.024	22
Apple	70	200	1.017	18
Wych Elm	70	180	1.018	16
Maple	77	145	1.018	20

The average production of three works in Paris gave from 4,000 pounds of beech and oak: Charcoal, 1,014 lbs.; Acid liquor, 2,335 lbs., sp. gr., 1.027,=dry acetate of lime, 416 lbs.; tar, 330 lbs.

When wood is distilled in close cylinders, the charcoal obtained is about 25 per cent. of the weight of the harder woods, as oak and beech, or about three-fifths of the total carbon present; the liquid part is about 53 per cent., containing about one-fifth of the carbon; the gases contain the remaining one-fifth of the carbon. Of the

liquid products, acetic acid equals about 3 or 4 per cent. of the harder woods, the naphtha about 1 per cent. and the tar 7½ per cent.

The time occupied in firing a charge is from twelve to sixteen hours; the latter time is preferable, since a slow carbonization yields a larger product. The amount of charcoal obtained depends largely on the length of time occupied. The following table, by Karsten, gives the results of some experiments upon air-dried timber :

SPECIES OF WOOD USED.	CHARCOAL.	
	By quick charging.	By slow charging.
Young oak	16.54	25.60
Old "	15.91	25.71
Young beech	13.12	25.22
Old "	13.65	26.45
Young alder	14.45	25.65
Old "	15.30	25.65
Young birch	13.05	25.05
Old "	12.20	24.70
Young deal	14.25	25.25
Old "	14.05	25.00
Young fir	16.22	24.72
Old "	15.35	24.75
Young pine	15.52	26.07
Old "	13.75	25.95
Lime	13.30	24.60

According to an English authority, the yield which should be obtained can be approximately calculated from the following data. One ton of wood, delivered at the works, will yield 2½ to 3 cwt. of bark, and, if fairly dry, 5 cwt. best charcoal, 1½ to 2 cwt. of best brown acetate, or 1½ of grey, 1¾ to 2 gallons of "miscible" naphtha at 60° over proof, or 2½ gallons of "solvent" naphtha at 45.°

Many improvements in the methods for wood distillation have been preposed from time to time, especially with reference to the utilization of wood in a finely divided state, such as spent residues from tanning and dyeing operations, saw-dust, &c. With properly constructed apparatus, the yield of acid is nearly as large as with fresh wood, but the charcoal, owing to its finely divided state, is almost worthless.

The use of hydrochloric acid for making a superior quality of acetate of lime is as follows: A clear solution of brown acetate is made and evaporated to one-half its bulk, removing the impurities

as they rise to the surface. When evaporated, hydrochloric acid is added until litmus paper is just reddened. The resinous products are thus separated and the volatile bodies driven off on further evaporation. About 5lbs. hydrochloric acid to every 41 gallons of liquid, after the naphtha has been expelled, are usually sufficient. The solution is then boiled down to dryness, giving a brown acetate of a remarkably pure quality.

This use of hydrochloric acid is not general in America. It has this disadvantage, that it leaves in the finished product a certain amount of chloride of calcium. When an acetate thus made is distilled with sulphuric acid to obtain the acetic acid the chlorine is carried over as hydrochloric acid and condenses with the acetic acid. We have tested many samples of American make of acetate of lime and find but very few which contain any chlorine at all; in every case where it existed in more than traces, the acetate had been made from shell-lime and hence the chlorine was present in the form of salt.

The lime used for making acetate should be as pure as possible and free from magnesia and silica. Both retard the slaking. The presence of 1 to 2 per cent. of magnesia is not specially injurious; at 10 per cent. the lime is perceptibly deteriorated and at 20 per cent. and upward it is too poor for use. Lime made from oyster shells is sometimes used. It contains more or less of salt, which is therefore carried through the various operations of mamufacture and appears in the finished product. The salt may vary in amount from one per cent. to six or even more in the acetate of lime. Its presence is injurious when sulphuric acid is used for distillation as previously mentioned under the use of hydrochloric acid in the manufacture. It is also liable to cause serious mistakes in the chemical analysis of acetate of lime when analyzed by distillation, unless specially guarded against. We shall refer to this later on when we come to speak of methods of analysis. A sample of stone lime which gave an acetate of low strength, was found to contain 5.49 per cent. of magnesia; another lime giving an acetate of a better quality, was found to contain but 1.35 per cent. of magnesia.

Having thus glanced at the materials which enter into the manufacture of this important article of commerce, we are prepared to discuss the different kinds of acetate of lime now manufactured and to call special attention to a few points which have much to do with its successful production.

Some years ago, three kinds of acetate of lime were made, the

black, the brown and the grey: but with improved methods of manufacture the black acetate has disappeared and but two kinds, the brown and the grey are now in use. The black acetate contained on an average about ten per cent. less acetate of lime than the brown.

In order to show the composition of representative samples of the brown and the grey acetates of lime, we give below two analyses, each of which is an average of twenty-six samples, taken at random from samples analyzed by us during the last three months. The reaction of both samples is alkaline, from the presence of caustic lime.

	Brown.	Grey.
Moisture, at 100° C.,	6.40	4.07
Insoluble, sand, &c.,	1.13	0.87
*Lime	23.95	29.29
Magnesia	0.62	0.15
†Acetic acid, anhydrous,	42.42	53.34
Tar, coloring matter, &c	25.48	12.28
	100.00	100.00
*By *English Commercial Test.*		
Equiv. to acetate of lime	67.37	82.64
†By *Distillation.*		
Equiv. to Glacial acid	49.51	62.32
" " Acetate of lime	65.19	82.05

The following tables of analyses include all the samples which have been analyzed by us during the last two years, ending April 30, 1882. The average of the brown acetates would be slightly increased if it included samples of a few makes of high-grade brown acetate ; but as these lots are usually sold without analysis they do not enter into the average given here. The analyses given below are a fair average of the general class of brown and grey acetates of lime now in the market.

Brown Acetate of Lime ;—*By English Commercial Test.*

No. of samples.	Moisture at 100° C.	Acetate of Lime.	Acetate of Lime (on dry basis.)
179	7.13	67.53	72.71

Grey Acetate of Lime ;—*By English Commercial Test.*

No. of samples.	Moisture at 100° C.	Acetate of Lime.	Acetate of Lime (on dry basis.)
84	5.33	81.01	85.57

Grey Acetate of Lime ;—*By Distillation.*

No. of samples.	Moisture at 100° C.	Glacial Acid.	Acetate of Lime.	Glacial Acid (on dry basis.)	Acetate of Lime. (on dry basis)
22	6.22	62.60	82.42	66.75	87.88

This table of grey acetates, analyzed by distillation, is composed of exceptionally high-grade samples. They are very light in color, containing but little tar and yielding an amount of acetate of lime above the general average of the grey limes quoted above. By the "English Commercial Test" the average of these twenty-two samples would show about 89.00 per cent. acetate of lime on dry basis.

The presence of *chlorine*,—either as *chloride of calcium* from the use of hydrochloric acid in the manufacture, or as *chloride of sodium* from the use of shell-lime——is not usual in American acetates of lime. Out of twelve samples, taken at random from nearly sixty samples analyzed by us during the last three months, ten samples showed no trace of chlorine, one contained a trace only and one had about two per cent. of chlorine. Both the latter samples were made from shell-lime.

In order to make an acetate of lime of as high a grade as possible, three results should be sought :

First : The removal of the *tar*.

Second : The finished product should contain as little *water* as possible.

Third : An undue *elevation of temperature* during the drying of the acetate should be most carefully avoided.

In an article which contains so large a percentage of a valuable ingredient as does acetate of lime, the presence of even one per cent. of impurity causes a loss of 0 7 to 0.9 per cent. acetate of lime ; that is, the one per cent. of useless matter, be it insoluble matter, tar or water, takes the place which might be occupied by valuable acetate of lime.

For example : Two lots of brown acetate of lime are taken, each containing originally 9 per cent. of water and 66 per cent. of acetate of lime. One lot remains unchanged ; the second, by exposure, dries so that it contains 6 per cent. of water. The acetate of lime is therefore raised to 68.17 per cent. The total weight of the second lot would be less than it was at first, but the amount of acetate of lime in one hundred pounds, that is, stated as a percentage would be raised to 68.17. Both lots taken each as a whole would contain exactly the same total amount of acetate of lime. One ton of 2,000 lbs. weighed out from the first lot, which remained unchanged, would contain 1320 lbs. acetate of lime ; but one ton of 2,000 lbs. weighed out from the second lot, which had dried, would contain 1363.4 lbs. acetate of lime.

So of two lots of grey acetate of lime, each containing at first 9 per cent. water and 80 per cent. acetate of lime. By drying to 6 per cent. water the second lot would contain 82.63 per cent. acetate of lime in each one hundred pounds of the dried-out acetate.

Since acetate of lime when dry rapidly absorbs moisture, a sample containing only 2 or 3 per cent. of water might absorb as much more by exposure in a damp place, and the amount of acetate of lime would be proportionally diminished when reckoned into percentage on one hundred pounds. The total weight of the lot would be increased, but the actual amount of acetate of lime in each one hundred pounds of the dampened lot would be less than in one hundred pounds of the lot when first made.

This brings us to the third requisite needed for a high-grade article, the *Drying of the acetate of lime.* It is here that care is indeed needed. A rise of temperature of only 50° Fah. above the safety point begins at once to decompose the acetate of lime and the product rapidly loses in strength with further increase of temperature. We have been unable to find any special reference to this important point in the manufacture of this article. It is indeed stated in books that great care is needed in the drying and that the acetate is easily injured by heat, being decomposed at about 460° Fah. But we have met with no record showing the rate of decomposition produced by length of time or increase of temperature. We have made a long series of experiments on this point, because of its importance, in connection especially, with the manufacture of acetate of lime. We sought to ascertain the temperature at which the decomposition begins, and also to see what influence, if any, the impurities possess in hastening such decomposition. We used for this purpose a so-called chemically pure, white acetate of lime,—which had lost by efflorescence some of its water of crystallization, a commercial brown acetate of lime—the average of twenty-six samples, the full analysis of which has been already given, and a commercial grey acetate. An analysis, by distillation, was first made. Then weighed quantities of the samples were submitted to heat for different lengths of time as detailed below, and the residues were then distilled and the acetic acid determined. The results are calculated to a dry basis for better comparison.

A preliminary trial with a sample of acetate of soda, showed that it was not at all decomposed by an exposure for one hour at 550° Fah.

ACETATE OF LIME—PURE.

	Glacial Acid.	Acetate of Lime.
Sample originally contained,	74.53%	98.13%
Heated for 1 hour, at 500° Fah.	74.35	97.89
" " " " 550° "	73.32	96.54
" " " " 600° "	72.08	94.90

The acetate after heating at the high temperatures above given, was strongly alkaline, from caustic lime. At 500° Fah. no change took place in the color; at 550° Fah. it was in places very slightly browned; at 600° Fah. it was but little darker in color than at 550° Fah. Chemically pure acetate of lime contains 75.95 per cent. of glacial acid, on a dry basis.

	BROWN ACETATE.		GREY ACETATE.	
	Glacial Acid. per cent.	Acetate of Lime. per cent.	Glacial Acid. per cent.	Acetate of Lime. per cent.
Sample originally contained,	52.89	69.64	67.09	88.33
Heated for ½ hour, at 400° Fah.,	52.77	69.48	67.09	88.33
" " " " 450° "	50.15	66.03	66.17	87.12
" " " " 500° "	50.15	66.03	64.86	85.40
" " " " 550° "	48.83	64.29	63.40	83.47
Heated for 1 hour, at 400° Fah.,	51.01	67.16	67.09	88.33
" " " " 450° "	50.16	66.04	65.89	86.75
" " " " 500° "	49.65	65.37	64.09	84.38
" " " " 550° "	48.83	64.29	63.09	83.07
" " " " 600° "	——.	——	61.94	81.55

We have here, we think, a clue to at least one of the causes which sometimes yield an acetate of lime far lower in strength than was expected by the manufacturer. We see by the above table that the greater part of the loss occurs during the first half hour of exposure at 500° Fah., that it is three times as great in the brown acetate as in the grey, and that for every additional 50° Fah., the loss will average about 1.40 per cent. of acetate of lime. In a substance so easily decomposed by a high temperature as is acetate of lime, we cannot expect that the results obtained will be in exact ratio to the increase of time or temperature. The figures are given just as obtained from the analyses. Several of them, over 500° Fah. are the results of duplicates, the greatest difference being one half of one per cent. for an hour's heating.

The limit of safety is 400° Fah., for the grey acetate, and 400° Fah. for one half an hour for the brown acetate. This temperature

will brown white paper by an exposure of fifteen or twenty minutes. An increase of fifty degrees up to 450° Fah., would not be readily noticed by the operative, but its results would surely be found in the poorer quality of the article produced.

When brown or grey acetates of lime are distilled in a current of steam the distillate is always more or less colored. But after exposure to a temperature of 450–500° Fah. for half an hour or so, the distillate is perfectly colorless. The tarry matters are no longer carried over, but this advantage is gained at a loss of a certain part of the acetate by decomposition, as shown above.

Many samples of acetate we analyze contain pieces or crusts which appear to have been melted or partly burned; some lots contain a large amount of such pieces and their presence must diminish to a certain extent the percentage of real acetate present. A sample of the grey acetate referred to in the table above, was heated for an instant until partly melted and the glacial acid was found to be 65.46 per cent. a reduction of 1.63 per cent., equivalent to 2.14 per cent. acetate of lime.

SAMPLING.

In drawing samples for analysis it is important to obtain a fair average of the lot. One bag in every ten is the usual number sampled; if the lot is small, one bag in every five or six is sampled, care being taken to obtain a fair proportion of both large and small pieces. The samples are taken from the bags by meams of a large cylindrical tryer and put at once into a rubber bag to protect them from change, either by absorbing or losing moisture. The average sample thus obtained is taken to the laboratory and prepared for analysis by thorough mixing and sifting until the final average is reached. The moisture of the sample, as drawn, is determined and is reported in the certificate of analysis.

The final average prepared for analysis may have lost or gained moisture during the operations of grinding, &c., hence it is necessary to make a correction of the analytical results obtained, in order to report the correct amount of acetate of lime present.

For example: a sample of brown acetate prepared for analysis contained 7.04 per cent. moisture, and 70.33 per cent. acetate of lime. But the sample as drawn from the bags contained 9.74 per cent. water; hence the result of 70.33 per cent. is corrected to 68.28 per cent. based as the 9.74 per cent. of water actually present in the goods. A sample giving 66.86 per cent. acetate of lime, with

7.00 per cent. of water in the prepared sample was corrected to 67. 66 per cent. based on 5.90 per cent. water present in the original sample. Some samples containing large amounts of tar or moisture need a preliminary drying of the sample selected for analysis; hence the correction to be applied is much larger than is usual, where the samples are easily ground and sifted.

It sometimes happens that samples are received for analysis, which come in paper boxes or wrappers. Such a sample will very seldom represent the actual condition of a lot of acetate as shipped from the factory. The paper box or wrapper offers but little obstruction to a loss of moisture by drying. Hence the sample received is very likely to give a much better result than will be obtained when the goods are received, and sampled from the bags for analysis.

The amount of moisture present should always be stated in the certificate of analysis of acetate of lime, because it is an important factor, showing the condition of the goods at the time of sampling. This determination of moisture must be made on the sample as drawn, before it has been subjected to any treatment which will at all affect the amount of moisture present.

METHODS OF ANALYSIS.

1.—*Based on the Amount of Soluble Lime salts present.*

The value of an acetate of lime depends entirely on the amount of glacial acetic acid present. Two methods of analysis are in use at the present time; one determines the amount of lime salts soluble in water, and by calculation the amount of lime so found is converted into acetate of lime, or glacial acid, as the case may be. This method is based on the supposition that all the soluble lime salts present are acetates; but this is not so. Acetates of lime almost invariably contain caustic lime in slight amount, and if the lime has been overheated it is present in still greater quantity. Again, organic salts of lime are always present. With improved methods of manufacture, the amount of these has diminished of late years, and the difference between the amounts of acetate of lime found by distillation of the acetic acid, and that found from the amount of soluble lime is much less than formerly. The better the sample of acetate, the less is the difference between the results given by the two methods.

In the year 1872 (see *American Chemist*, vol. II, p. 324, and vol. III, p. 8), this question of methods of analysis came up. It is un-

necessary to state here the objections raised by certain English chemists against the process of distillation, which was advocated by some American chemists, since the objectors to a distillatory process had no good foundation upon which to rest. But as the general custom of the trade was at that time based upon the method of analysis by means of the soluble lime salts present, we have before and since that date used the term "*English Commercial Test*," to designate analyses made in this manner, and so state the results on our reports of analysis. Whenever an analysis is made by distillation it is so stated.

2.—*Based on the Distillation of the Acetic Acid.*

Three acids may be used in the process of distillation—hydrochloric, sulphuric or phosphoric.

a.—When *hydrochloric acid* is used, a part of it is carried over with the acetic acid and must be estimated and a correction made. It possesses these two advantages: that it does not act upon the organic matter present, and that the solution of chloride of calcium will permit of the distillation being carried to a low point without danger of error. The distillation is made in a retort connected with a condenser, and the total acids present in the condensed liquid are estimated, and the proper correction applied for the amount of hydrochloric acid found to be present.

b.—The use of *Sulphuric acid* has three disadvantages.

First.—As it becomes concentrated in the retort during the process of distillation, it acts upon the organic matter present, forming sulphurous acid, which is carried over with the acetic acid and included in the estimation of the total acid power of the distillate. Thus the amount present would be calculated as acetic acid, and the result thereby increased unduly. To prevent such action, recourse is had to distillation in a current of steam. (See American Chemist, vol. vi. p; 294.)

Second.—The sulphate of lime formed by the addition of sulphuric acid to the solution of the acetate of lime, is troublesome on account of the bumping which takes place during the distillation. This is partly prevented by the use of a current of steam.

Third.—When a sample of acetate of lime contains *chloride of calcium* or *chloride of sodium*, the chlorine is carried over in the form of hydrochloric acid and neutralizes its equivalent amount of the standard soda, used to receive the acid distillate. The amount thus distilled must be estimated and the correction made. To prevent this, the chlorine present is precipitated by the addition of

sulphate of silver to the solution of acetate before distillation begins.

c. Phosphoric Acid is the best acid for use in the process of estimating acetic acid by distillation. It has three advantages.

First.—It does not act on the organic matter.

Second.—During the distillation, the liquid in the retort is not suffered to fall below 15 c.c. in bulk. Under such circumstances, phosphoric acid does not decompose any chlorides of calcium or sodium which may be present in the acetate, thus requiring no addition of sulphate of silver. Only the merest trace of chlorine is carried over, as shown by experiments with a sample of acetate containing nearly 5 per cent. of chloride of sodium and also by trial on a known amount of pure chloride of sodium. This unexpected result merits attention, since in this respect phosphoric acid differs so decidedly from sulphuric acid.

Third.—It forms a perfectly clear solution with the acetate of lime. By the use of a large amount of glacial phosphoric acid, at least five times the weight of acetate of lime taken for analysis, the phosphate of lime first formed is redissolved to a clear solution. The distillation proceeds quietly and uniformly without bumping and the amount of liquid in the retort is easily kept at any desired point. The use of a current of steam is necessary both for ease of manipulation and accuracy of results.

After a thorough trial of hydrochloric and sulphuric acids, we discarded both in favor of phosphoric acid and for some years past have used it to our full satisfaction. It is important that the phosphoric acid used should contain no nitric or other volatile acids, which would increase the results obtained above the truth. Each new lot of phosphoric acid should be examined for such impurities before use. If it be suspected that any phosphoric acid has been carried over during the distillation, it is easily detected in the distillate by the use of molybdate of ammonia.

The process of distillation, if carefully and intelligently done, is very accurate. Duplicates made by this method agree with each other fully as closely as do those made by the estimation of the soluble lime. For example: In January last, a sample of grey acetate was analyzed by distillation in our laboratory, with a result of 78.22 per cent. acetate of lime. Three months later, the same sample was reanalyzed by another man in our laboratory, using fresh standard solutions, and 78.20 per cent. of acetate was obtained. This sample contained 3.16 per cent. of common salt,

shell-lime having been used in its manufacture. See also the table of analysis quoted above under "Drying of the Acetate of Lime." With care and experience in this method of analysis by distillation, it is not difficult to obtain duplicates which agree to two tenths of one per cent. of acetate of lime; usually the difference is less than that. A sample of grey acetate analyzed recently, was distilled with sulphuric acid and a duplicate with phosphoric acid. The results agreed exactly; the latter distillation, however, requiring less attention than the former, for reasons given above under processes of distillation.

The indicator used for titration is a few drops of a solution of phenol-phthalein—one gramme in 250 c.c. of a mixture of equal parts or water and alcohol. We have found this indicator far preferable to either litmus or cochineal; of course, whichever indicator is used, the same must be used both for the standardizing and the actual analysis.

To show the need of some uniform and accurate method of analysis, we refer to a note in Allen's Commercial Organic Analysis, vol. I. p. 205. He finds from experiments made in his own laboratory on the *same sample* of acetate of lime, that results were obtained varying from 47.4 per cent. to 57.6 per cent. of acetic acid. In our own practice for many years previous to the publication of this book we found differences fully as great between results by distillation and by the various commercial processes; but, as we said before, at the present time the differences are not nearly so great as formerly.

In conclusion we wish to lay strong emphasis upon the facts, that, inasmuch as the process of distillation is the only one which gives the real amount of glacial acid present, and, inasmuch as it is an imitation of the actual manufacturing process for obtaining acetic acid from its acetates, it is the most reliable and should be adopted. This position we have maintained for many years, and we notice that, especially in the case of grey acetate, buyers are more and more insisting on the test by distillation. The price should be based on the unit of glacial acid, just as in fertilizing materials, the prices are based upon the units of phosphoric acid nitrogen and potash.

FOREIGN PATENTS.

FARBWERKE, successor to MEISTER LUCIUS & BRUNING, Hoechst: *Method of preparing coloring matters by the action of the disulphonic acids of B-naphthol upon the diazo compounds of aromatic acids.* (Germ. P. 15,250, Jan. 22, 1881.) Red dyes result from the action of the two disulphonic acids of B-naphthol upon the diazo-compounds of ethylic and methylic ethers of aromatic acids. The methylic and ethylic ethers of para-diazocinnamic acid yield red, those of diazo-a-naphthoic acid bluish red, and those of diazo-B-naphthoic acid claret red coloring matters.

O. N. WITT, Muelhausen: *Method of preparing coloring matters by the action of nitroso-compounds or chloroquinoneimides upon aromatic metadiamines.* (Germ. P. 15,272, Nov. 6, 1880.) By the action of the nitrosoderivatives of tertiary aromatic amines, or of nitrosophenols upon metadiamines, particularly upon phenylene and toluylene-diamine coloring matters result which upon oxidation yield new coloring matters. Instead of nitroso-compounds the so-called quinonechlorimides, obtained by allowing solution of chloride of lime to react upon hydrochloride of amidophenol or upon the chlorhydrates of paradiamines, may be employed. These yield blue and violet coloring matters which change to red upon boiling.

R. IRVINE, Royston Glanton: *Improvements in the manufacture of lubricants.* (Germ. P. 15,397, Nov. 24, 1880).

F. HEEREN, Hanover; *Method and instrument for testing milk.* (Germ. P. 15,681, Feb. 22, 1881). A disc upon which are six radial panels of different shades, the colors of which correspond to the appearance of milk of different degrees of fatness. There is also a dark central panel. Upon this a thin layer of the milk to be tested is produced. The panel corresponding in color with this layer shows the degree of fatness of the milk.

C. SCHEIBLER, Berlin: *Method of preparing saccharate of strontium from molasses and syrup.* Germ. P. 15,385, July 24, 1880). Tribasic saccharate of strontium separates at a boiling heat from the above liquid, and is purified by washing with hot water. At a lower temperature the strontium saccharate so obtained is decomposed into a less basic saccharate and free strontium hydroxide. The former is used for defecating beet juice and similar liquids.

KUNHEIM & Co., Berlin: *Method of removing gypsum from solution of glucose obtained by the aid of sulphuric acid.* (Germ. P. 15,076, Jan. 29, 1881). Barium oxalate or phosphate is added to the solution after neutralization of the sulphuric acid with chalk. Instead of the above, any other barium salt the acid of which forms an insoluble compound with lime may be used.

P. HOSEMANN, Berlin: *Sizing for woven goods, wall paper, calico printing, felting &c.* (Germ. P. 15,251, Feb. 1, 1881). A size unalterable, odorless and neutral is prepared by mixing solution of calcium chloride, potato starch, water, rosin dissolved in potash and soda lye, and heating to 140–170°F. A mixture of "cream of tartar preparation," sulphuric acid and solution of aluminium sulphate stirred up in water, is then added. Finally solution of zinc chloride, water, phenol and nitrobenzole. Some of the above substances may be left out or others substituted to suit the purpose for which the sizing is intended.

H. SCHMIDT, Frankfort on M: *Method of preparing nitrobenzaldehyde from nitrobenzylchloride with the aid of metallic oxides.* (Germ. P. 15,881, Feby. 20, 1881). Paranitrobenzylchloride is heated to 200–250°C. with 1–2 parts oxide of copper or peroxide of lead. Besides the above mentioned substance, nitrobenzylbromide, sulphide and hydrosulphide can be converted into nitrobenzaldehyde.

A. P. DUBRUNFANT, Paris: *Manufacture of maltose.* (Engl. P. 1,048, March 11, 1881). Extract of malt is added to a dough of starch and warm water. The mixture is kept at a high temperature until the dextrine is all converted into maltose. The resulting liquid is clarified, filtered and evaporated *in vacuo.*

B. ROEBER, Dressle: *Disinfection and clarification of sewage and waste waters from industrial establishments.* (Germ. P. 15,392. April 25, 1879). Addition of lime and tar. Waters difficult to clarify receive besides the above a quantity of magnesium chloride.

C. V. CLOLUS, Paris: *Obtaining substances from soap boilers' waters.* (Eng. P. 681, Feb. 16, 1881). Neutralization with HCl evaporation to 32° Bé. to separate salt, and treatment of the liquid portion with hot air to remove water from the glycerine.

H. STIER, Gerstewitz: *Arrangement for rendering the hot waste gases from coke furnaces suitable for certain chemical purposes.* (Germ. P. 15,683, May 23, 1880). The heat of the gases is used for heating a cylinder containing coke in which water gas is generated. The latter, together with part of the above furnace gases, are employed in heating soda furnaces, etc.

A. ROSSI and L. BECKWITH, New York: *Method of generating cold.* (Germ. P. 15,151, Jan. 18, 1881). An ammonia ice machine in which the ammonia is dissolved in glycerine.

J. S. BARFF, London: *Preservative for organic matters.* (Engl. P. 1,33[illegible], March 25, 1881). Use of boracic acid dissolved with the aid of heat in glycerin.

E. CHESNAY, Paris: *Depilatory for hides.* (Germ. P. 15,736, Nov. 6, 1880). Solution of ammonium sulphite.

W. WOLTERS, Kalk, near Cologne: *Method of applying an*

acid-proof coating to iron vessels used for distilling sulphuric acid. (Germ. P. 15,639, Nov. 11, 1880). By heating a mixture of pyrosulphates of the alkalies and concentrated sulphuric acid in iron vessels, the latter are said to become coated with sulphide of iron, and to be acid-proof at the distilling temperature of sulphuric acid provided the acid sodium sulphate be left in the vessels during the distillation.

E. Solvay, Brussels: *Method of calcining the sodium bicarbonate obtained in the ammonia soda process.* (Germ. P. 16,131, May 10, 1881). Adds a certain quantity of calcined soda to the bicarbonate to prevent the latter from becoming pasty in the calcining apparatus.

E. Solvay, Brussels: *Method of utilizing native basic phosphates by employing them in the manufacture of soda and potash by the ammonia soda process.*(Germ. P. 16,229, April 15, 1881). The phosphates are concentrated in the following manner: They are first pulverized and washed in order to separate part of the lighter calcium carbonate. The powder is afterwards calcined whereby the lime not combined with phosphoric acid becomes causticized, it is then employed in decomposing the ammonium chloride resulting from the ammonia soda process. Calcium chloride dissolves out and calcium phosphate containing but little excess of lime remains.

H. Koechlin, Loerrach and O. N. Witt, Muelhausen: *Preparation of blue and violet dyes.* (Germ. P. 15,915, Mar. 19, 1881.) The coloring matters can be obtained by two different methods.

I. The nitroso derivatives of tertiary aromatic amines or phenols, as well as the so-called chlorquinoneimides, yield coloring matters if allowed to remain in contact with alkaline or ammoniacal solutions of phenols at the ordinary temperature. Their formation can be effected immediately if the re-action be initiated by the addition of reducing agents, as zinc dust, stannous oxide or grape sugar.

II. The same coloring matters are formed if weakly alkaline, neutral or weakly (preferably acetic) acid mixtures of phenols, with para-amido bodies of phenols, primary, secondary or tertiary aromatic amines be treated with oxidizing agents. As such atmospheric oxygen, chromates, ferricyanides, permanganates, etc., may be employed.

Instead of preparing the coloring matters they may be produced directly upon the fibre, a proceeding which possesses certain advantages in calico printing. Three processes for doing this are described in the patent. (See also this Journal vol. IV).

L. Wollheim, Vienna: *Method of obtaining pure caustic alkalies by means of electrolysis.* (Germ. P. 16,126, Apr. 13, 1881.) A diaphragm divides the decomposing vessel into two chambers. In the one is placed the negative, in the other the positive electrode. At starting a solution of the alkali to be prepared is poured into

the former, into the latter a solution of the salt to bendecomposed, carnallite for instance. If the electric current does ot exceed a certain strength a continuous flow of caustic potash solution, uncontaminated with salt solution can be obtained.

A. HEGENER, Cologne : *Improvements in the fractionated distillation of coal.* (Germ. P. 14,889, Oct. 17, 1880.) The object of this invention is to obtain illuminating gas and fuel gas from the same charge of coal, the latter gas being chiefly evolved at the beginning and towards the end of the distiilation. For this purpose the retort furnaces are provided with two separate mains and the dip pipes of both furnished with valves.

C. A. BURGHARDT, Manchester : *Treatment of animal and vegetable fats for the manufacture of soap.* (Eng. P. 5,191, Dec. 11, 1880.) The crude fatty matters are heated to a 130—180° in a retort by means of high pressure steam. As soon as vapors begin to form, a stream of cold or warm air is blown upon the surface of the fatty matters. The distilling fatty acids are absorbed by water or caustic lye.

H. P. HOSEMANN, Berlin : *Finish for woven fabrics.* (Germ. P. 16,110, Nov. 15, 1880.) The fabrics are passed through a solution of silk, wool or feather-down in caustic soda, then through a bath of sulphuric acid and finally through water.

ACTIEN—GESELLSCHAFT FUER ANILINFABRIKATION, Berlin: *Manufacture of orange red, red and brown—red azo dyes from methylnaphthalene.* (Germ. P. 15,649, Feb. 18, 1881). That part of coal tar which boils between 225° and 250° C. consists essentially of methyl and dymethylnaphthalene. From these, by nitration, conversion into the sulphonic acids and reduction amidomethyl (or, as the case may be, dimethyl) naphthalenesulphonic acid is prepared. The diazo compound of this acid yields coloring matters with betanaphthol or its sulphonic acids.

Or methylnaphthalene is transformed into the sulphonic acid and subsequently by fusion with potassa into methylnaphthol. This body yields with diazosulphanilic acid, or with diazonaphthalenesulphonic acid, or with amidoazobenzenedisulphonic acid yellowish red dyes; its sulphonic acid yields red dyes with the diazo-hydrocarbons.

ACTIEN GESELLSCHAFT "VIEILLE MONTAGNE," Angleur near Liege: *Manufacture of zinc white from solution of zinc chloride or sulphate.* (Germ. P. 15,249, Dec. 17, 1880). Excess of ammonia is added to the solutions so as to redissolve the zinc hydroxide. Ferric and manganic oxide remain undissolved and can be separated by filtration. Steam is then blown into the filtrate, to distill off the excess of ammonia and to precipitate the zinc hydroxide. The latter is filtered off, dried and heated. The ammonia is recovered from the solutions of sulphate and chloride by adding lime and distilling.

C. SEMPER, Philadelphia: *Manufacture of aluminium sulphate.* (Eng. P. 2,997, July 7, 1881). Precipitates iron from the solution by adding potassium ferrocyanide and cupric sulphate.

BADISCHES, GESELLSCHAFT FUER ZUCKERFABRIKATION, Waghæusel: *Improvements in the method of dry distillation of molasses swill for the purpose of obtaining swill char, ammonia, methylic alcohol and combustible gases.* (Germ. P. 15,702, Dec. 4, 1880). The evaporated swill is subjected to dry distillation with 5 to 15 per cent. of the dry substance of KOH obtained by causticizing with lime a portion of the char from a previous operation. The nitrogenous constituents of the swill are decomposed into simple ammoniacal compounds and a contamination of the potassium carbonate with lime avoided.

HARING, EHRENBERG & Co. and M. BASWITZ, Halle $^{o}/_{s}$: *Improvements in the dry distillation of molasses swill, elution lyes and osmose waters for the purpose of obtaining ammonia.* (Germ. P. 15,751, April 2, 1881). The liquid, concentrated to sp. gr. 1.35–1.39, is allowed to fall in drops into retorts heated to redness. The resulting gases are made to pass through a number of heated tubes located in the same furnace, in order to decompose the difficultly manageable tar, before they enter the receiver.

H. OPPERMANN, Bernburg: *Method of converting molasses swill into a dry powder with the acid of caustic lime.* (Germ. P. 16,033, Nov. 21, 1880).

XVII. ON THE DETERMINATION OF REVERTED PHOSPHATES.*

BY THOMAS S. GLADDING, A. M.

In an investigation on the subject of reverted phosphoric acid, the first question that arises is, What is meant by this term? The curious phenomenon of a decrease in the amount of soluble phosphate in a superphosphate, and its reversion into a form insoluble in water, but soluble in the organic salts of ammonia, in weak acids and other reagents, has been attributed to several different causes.

According to the most common explanation,† it is due to the gradual interaction between the soluble or monocalcic phosphate formed in the manufacture, and the still undecomposed tricalcic phosphate of the original rock, this reaction resulting in the formation of the intermediate compound dicalcic phosphate.

Again, it has been noticed that this reversion is especially large when the amounts of iron and alumina present are considerable, and in such cases it has been attributed‡ to the formation of phosphates of iron and alumina having the solvent properties specified above.

Again, from experiments in our own laboratory, it has been found that this reversion is very noticeable in acid phosphates, which, after treatment with sulphuric acid, have been mixed with natural phosphates containing large amounts of carbonate of lime. The reaction that takes place between the monocalcic phosphate and carbonate of lime probably results in the formation of either the dicalcic or tricalcic phosphate. This latter salt, though insoluble in water, is readily taken up, on account of its fine state of division, by the reagents used for the estimation of reverted phosphates.

From these different explanations of the character of reverted phosphate, it is evident that the term is not capable of a strict chemical definition. It may consist in any given case of one or of several chemical compounds. The best definition that can be given is the general one condensed into the name itself. Reverted phosphates are those which, originally insoluble, but having been made soluble, have reverted or returned to the insoluble condition. They must now, however, be looked upon as:

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*Read before the American Chemical Society, March 17, 1882.

†Zeit. fur analyt. Chem. 10, Fresenius.

‡Zeit. fur analyt. Chem. 19, Z. Meyer.

(1.) Just over the border line that separates the soluble from the insoluble, lying very close to the soluble, and being brought back by a slight addition to the solvent power of water ; and, as

(2.) Widely separated and different in solubility from the insoluble form which they originally possessed. Like the flats lying just above the river's edge, they must be wholly covered by a slight rise in the water that affects the banks but little.

The object of the inquiry here described has been to establish the above distinctions, and to find a method of separation that shall in the actual analysis recognize them as nearly as possible. The course pursued has been that followed in similar inquiries :

I. An examination of the solubility of the different natural phosphates used in the manufacture of superphosphates.

II. An examination of the solubility of the different chemical compounds of which reverted phosphates have been found to consist ; and

III. An attempt based upon the results of these two inquiries to find the best method of separating these two classes.

In the examination of the natural phosphates, five different forms were taken as typical of all. These were :

1. *Apatite*, as type of the nearly pure tricalcic form in its most insoluble condition.

2. *South Carolina rock*, containing besides tricalcic phosphate, phosphates of iron and alumina, and carbonate of lime.

3. *Bone Ash*, the great organic source of phosphate.

4. *Navassa rock*, typical on account of its large percentage of iron and alumina ; and finally,

5. *Curacao phosphate*, as type of the island phosphates, notable for their peculiar mechanical condition and greater solubility in the organic salts of ammonia.

In examining the solubility of these natural phosphates, attention was first given to the effect of different relative proportions of phosphate and solvent. The standard method of Fresenius, Luck and Neubauer* was followed, using 50 cc. of citrate of ammonia solution, of sp. gr. 1.09, and made as nearly neutral as possible ; time, 30 minutes, temp. 35° C. Two series of experiments were made. In the first, one gram of substance was taken ; in the second, $\frac{1}{10}$ of a gram.

*Zeit. fur analyt. Chem. 10.

	SERIES I. P_2O_5 dissolved.	SERIES II. P_2O_5 dissolved.
Apatite	0 40 per cent.	1.54 per cent
South Carolina rock	2.00 "	6.60 "
Bone Ash	2.60 "	8.30 "
Navassa	2.65 "	10.10 "
Curacao	4.40 "	16.00 "

Comparing these tables, we see at once the fact that has been commented upon very frequently of late by German chemists as regards superphosphates, and to which special attention has been called within a few months by a printed letter of Dr. S. W. Johnson, in the case of Curacao phosphate, namely, that the percentage of phosphate dissolved increases very largely with the increase of solvent, or, what is the same, the decrease of substance taken for analysis. These tables show that this is true not only of reverted phosphates as found by the former, nor of Curacoa phosphate as shown by the latter, but also of all natural phosphates, including even the difficultly soluble apatite. This fact has a very important bearing upon the error involved in the determination of reverted phosphoric acid by this method, *i. e.*, an error as regards the strict separation of the reverted phosphates from the insoluble phosphates. Thus, supposing five superphosphates after the extraction of the soluble to contain no reverted phosphate, but to contain 10 per cent. of the above raw phosphates respectively, corresponding to about 3 per cent. of insoluble phosphoric acid, the citrate would attack these raw phosphates, and instead of the error being 0.04, 0.20, 0.26, 0.26 and 0.44 per cent. as would naturally be supposed from Series I, it would in reality be 0.15, 0.66, 0.83, 1.01 and 1.60 per cent. as shownby Series II. Thus, the superphosphates would be reported as containing these latter amounts of reverted phosphoric acid, when in fact they contained nore whatever.

This same fact of increased solvent power obtained by increasing the amount of solvent salt was shown to be true also of oxalate of ammonia, by treating 2 grams of Navassa rock and Curacao phosphate with 1, 5, and 10 grams of the salt dissolved in 100 cc. of water, at a temperature of 100° C. for 30 minutes.

	SERIES III.		
	1 gram.	5 grams.	10 grams.
Navassa, P_2O_5 dis.,	2.20	8.75	12.15
Curacao, " "	2.50	12.30	17.60

The influence of increase of temperature upon the amount of rock dissolved was shown by treating one gram of the different phosphates with 100 cc. of water containing four grams of oxalate at 20°C. and at 100°C.

SERIES IV.

	Apatite.	So. Car. Rock	Bone Ash.	Navassa.	Curacao.
At 20°C. P_2O_5 dis.	0.36 pr ct.	2. 25 pr ct.	2.72 pr ct.	2.85 pr ct.	5.51 pr ct.
" 100° " "	2.16 "	5.80 "	8.78 "	9 40 "	15 30 "

In performing the above experiments at 100°C. a strong odor of evolved ammonia was observed, and it was also found that the filtrates were decidedly acid in reaction. The importance of this and its bearing upon the subject will be better seen further on.

The results thus far given show very clearly the cause of much of the confusion that has arisen over the determination of reverted phosphates. This lies in the great facility with which the citrate and oxalate solutions attack the insoluble phosphate, especially when the proportion of solvent is increased or when the temperature of digestion is raised. Many chemists have been led by these difficulties to seek a better method. The suggestion has frequently been made that a weak or dilute acid be employed as a substitute for the organic salts of ammonia. Thus Grupe and Tollens* recommend the use of a $\frac{1}{4}$ per cent. of citric acid solution. The solvent power of this acid of $\frac{1}{4}$ per cent., also of oxalic acid of $\frac{1}{4}$ per cent. and $\frac{1}{10}$ per cent., also of hydrochloric acid of $\frac{1}{100}$ normal

*Berichte der deutsch, chem. Gesellschaft, 14, 754.

strength, upon the natural phosphates was examined. 100cc. of each of the above solvents were taken, the temperature of digestion was 40°C., time one hour. One gram was taken with each acid, and in addition also $\frac{1}{10}$ of a gram with the citric acid of $\frac{1}{4}$ per cent.

SERIES V

Acid employed—	Citric ¼ per ct.		Oxalic ¼ per ct.	Oxalic $\frac{1}{10}$ per ct.	HCl $\frac{1}{100}$ normal.
	1 gram.	$\frac{1}{10}$ gram.	1 gram.	1 gram.	1 gram.
Apatite, P_2O_5 dis.	0.74 per ct.	2.70 per ct.	3.20 per ct.	1.08 per ct.	2 56 per ct.
So. Car. Rock, "	2.80 "	11.40 "	8.75 "	2.68 "	4 25 "
Bone Ash, "	3.80 "	11.80 "	9.23 "	4.15 "	3.85 "
Navassa, "	3.30 "	14.00 "	9.96 "	3.50 "	4.15 "
Curacao, "	4.20 "	19.80 "	9.12 "	4.00 "	3.35 "

Supposing a fertilizer to contain 10 per cent. of insoluble rock, the citric acid would make the reverted phosphoric acid 0.27 per

cent., 1.14 per cent., 1.18 per cent., 1.40 per cent., 1.98 per cent. respectively in excess of the actual amount present. The excess thus introduced would be considerably greater than that caused by the solvent action of the citrate of ammonia solution, *vide* Series II., and the claim made for the citric acid solution by Grupe and Tollens that it dissolves nearly the same amount of phosphate from different quantities of substance taken for analysis shown to be wholly unfounded as regards its action on raw phosphates. (Compare columns 1 and 2 above.)

The action of the different solvents upon the dicalcic and tricalcic salts was next studied. These salts were obtained by precipitation, and on analysis found to agree so closely with the theoretical composition as to show the admixture of traces only of other forms. After thorough washing they were mixed with about an equal weight of calcium sulphate, in order to imitate the conditions under which they are found in a superphosphate. These mixtures were then thoroughly air-dried. One gram of each was now treated with the different solvents, already employed, for 30 minutes at a temperature of 40°C. The mixture of the dicalcic salt contained 19.20 per cent. of P_2O_5, of the tricalcic salt 13.00 per cent.

SERIES VI.

Solvent.	$2CaO,P_2O_5$.	$3CaO,P_2O_5$.
100 cc. ½ per cent. citric acid, P_2O_5 dis. =	10.60 9.31 per cent.	10.26 per cent.
100 cc. $\frac{1}{10}$ " oxalic acid, "	5.32 "	5.10 "
" $\frac{1}{100}$ normal HCl " "	3.42 "	1.90 "
50 cc. cit. of ammonia sol. "	19.20 "	13.00 "
50 cc. H_2O+1 gram oxal. ammo. "	19.00 19.05 "	11.50 "

It will be seen from this table that the citrate solution dissolves both forms perfectly; that the citric acid, though fairly successful with the tricalcic salt, dissolves only one-half of the dicalcic salt; that the other acids, notably the mineral acid HCl, fall far below the citric in this respect. Special attention is called to the vigor of the weak solution of oxalate of ammonia. Though only one gram is used, it dissolves all but a trace of the dicalcic salt, and falls but little short of dissolving all the tricalcic form. In the original article of Fresenius, Luck and Neubauer, the weak acids were discarded on account of their inability to take up the reverted phosphate. This conclusion is corroborated by these experiments, with the additional objection proved by Series V., namely, the greater extent to which they dissolve the insoluble phosphate. They found

also that oxalate of ammonia solution dissolved the dicalcic phosphate equally with the citrate salt, but abandoned its use on account of the vigor with which it attacked the raw phosphates. But they employed in their published experiments a very large amount of oxalate, six grams of the salt being taken. But it is here found that a much weaker solution of the oxalate is effective for dissolving the dicalcic salt. As to its solvent action upon the natural phosphates, a series of experiments proved that it was not as energetic in this respect as the citrate of ammonia solution, dissolving considerably less than the latter. On account of the many advantages possessed by an oxalate of ammonia solution over the citrate solution (1) as regards ease of preparing a perfectly uniform, neutral solution, (2) as to the greater facility of filtering and washing, and many others, many chemists urged and secured its adoption at the Cincinnati convention of agricultural chemists of 1881. But further experiments, to be described later, reluctantly compelled the conclusion that it cannot supersede the citrate of ammonia solution.

A series of experiments were now made upon a sample of acid phosphate, in order to compare the results obtained by the different methods, and to throw light, if possible, upon the causes of discrepancy. A sample made from Navassa rock was chosen on account of the large amount of reverted phosphate usually present in this class of fertilizers. The total phosphoric acid in the sample was 17.00 per cent., the soluble was 5.00 per cent., leaving 12.00 per cent. of reverted and insoluble. After thoroughly washing out the soluble, one gram was treated with 50 cc. of citrate of ammonia solution, and the directions of Fresenius carefully followed. Another gram was treated in exactly the same way, using however as a solvent 50 cc. of water containing in solution one gram of oxalate of ammonia. The following results were obtained:

SERIES VII.

	P_2O_5 dissolved.	P_2O_5 undissolved.
By citrate solution or "Washington method,"	6.10 per cent.	5.90 per cent.
By oxalate solution,	6.15 "	5.85 "

These two methods give results that are gratifying as regards their agreement. Subsequent experiments, however, showed conclusively that both were far from the truth.

Two grams of the same sample were now, after the extraction of

the soluble phosphate, treated according to the method adopted by the agricultural chemists at Cincinnati, known as the "Cincinnati method." The two grams of residue were washed from the filter paper into a breaker with 100 cc. of water, containing in solution two grams of oxalate of ammonia. (This is the same amount of oxalate in proportion to the quantity of substance taken as is used in the above determination.) 400 cc. of water were brought to boiling, and the breaker, closely covered, now introduced into the boiling water, the lamp being at the same time removed. The temperature of the liquid in the beaker rose to about 70° C. in a few minutes, and then slowly sank during an hour to about 40° C. The beaker was quickly rotated at intervals. Two grams were treated in exactly the same manner, using however, instead of the oxalate solution 100 cc. of the citrate solution.

SERIES VIII.

	P_2O_5 dissolved.	P_2O_5 undissolved.
By oxalate of ammonia,	8.55 per cent.	3.45 per cent.
" citrate "	9.85 "	1.15 "

The point of interest connected with these results is the much larger amount of phosphoric acid dissolved by both the reagents as compared with that dissolved by them at the temperature of 35° C. continuous. This is especially the case with the citrate solution, the dissolved phosphoric acid rising from 6.10 per cent. to 9.85 per cent., an increase of 3.75 per cent. This increase can be explained in two ways only: (1) Either the insoluble rock present is dissolved to this increased extent, or (2) there is a form of reverted phosphate present that is not fully taken up by either of the citrate or oxalate solution at the lower temperature.

In order to ascertain if the former of these two explanations was the correct one, two separate portions of $\frac{2}{5}$ of a gram each of Navassa rock, containing 0.120 gram P_2O_5, and corresponding to 6 per cent. of insoluble phosphoric acid when calculated on two grams as used in the above analysis (which is just the amount of apparently insoluble acid left in Series VII.), were rubbed to a paste in a mortar and treated each with 100 cc. of citrate of ammonia solution, the one at a temperature of 35° C. continuous for one hour, the other at a temperature falling from 70° C. to 40° C. during an hour, as in Series VIII. The following amounts of P_2O_5 were dissolved, calculated as per cent. on 2 grams:

SERIES IX.

No. 1.	Phos. acid dissolved,	1.16 per cent.
No. 2.	" " "	1.59 "

The increase in phosphoric acid dissolved by the use of the higher temperature is found to be only 0.43 per cent. It is seen at once that this small increase cannot explain the large increase of 3.75 per cent. in the case of the acid Navassa, made from the same rock.

The second of the two possible explanations, viz.: The presence of a form of reverted phosphate not fully dissolved at 35° C., must therefore be the true one. The experiments already made show that this form can be neither the dicalcic nor tricalcic phosphates, as the citrate solution is a perfect solvent for these two forms at a temperature of 35° C. The only form left is the reverted phosphates of iron and alumina. A quantity of the mixed normal phosphates of iron and alumina, Fe_2O_3, P_2O_5 and Al_2O_3, P_2O_5, was now prepared by precipitation. A portion was mixed with a large quantity of calcium sulphate and dried to a powder at a temperature not exceeding 60° C. The remainder was dried at the same temperature without admixture. One gram of the first portion containing 4 per cent. of phosphoric acid was now rubbed to a paste in a mortar and then digested with 100 cc. of the oxalate and citrate solutions, first at 35° C. and then at 70° C. to 40° C. for one hour.

SERIES X.

	P_2O_5 dis. at 35°C.	P_2O_5 dis. at 70° to 40°C.
Using oxalate of ammonia,	0.95 per cent.	3.20 per cent.
" citrate "	1.60 "	4.00 "

One gram of the unmixed portion containing on analysis 35.62 per cent. of phosphoric acid was now treated exactly in the same way, with results as follows :

SERIES XI.

	P_2O_5 dis. at 35°C.	P_2O_5 dis. at 70° to 40° C.
Using oxalate of ammonia	11.06 per cent.	26.50 per cent.
" citrate "	13.14 "	35.62 "

These two series of experiments prove the very important fact that neither the citrate nor oxalate solution will dissolve more than a small portion of the reverted phosphates of iron and alumina at the temperature of 35° C., and that even at the higher temperature

of 70° C. to 40° C. the oxalate falls short of the perfect solvent power which must be required of it.

These results explain most clearly the great increase of phosphoric acid taken up in Series VIII. over that dissolved in Series VII. The acid Navassa under treatment contains a very large quantity of reverted phosphates of iron and alumina which are not dissolved at the lower temperature. They also explain why the citrate solution dissolves in 1.30 per cent. more than the oxalate, because of its more vigorous solvent action on these forms of reverted phosphates. One gram of these phosphates of iron and alumina, treated with 100 cc. of the citrate solution at a continuous temperature of 65°C. for 30 minutes, formed also a clear solution.

In order to ascertain the amount of solvent action of this method upon the different forms of raw phosphates, $\frac{1}{5}$ of a gram of each, corresponding to about 3 to 4 per cent. phosphoric acid when calculated upon two grams, was treated in the same manner as the above, temperature 70°C. to 40°C., using 100 cc. citrate solution.

SERIES XII.

	P_2O_5 dis.
Apatite	0.22 per cent.
South Carolina rock	0.61 "
Bone Ash	....
Navassa rock	1.09 "
Curacao	2.07 "

Comparing this table with Series II., dividing the results there by by ten, it is evident that at the higher temperature, only a small increase in the amount of insoluble rock dissolved is observed.

In this connection special attention is called to one point. Notice has been called to the fact that at 100°C. the oxalate solution loses ammonia and becomes decidedly acid in reaction. A solution of neutral oxalate, immersed in boiling water for one hour in an open beaker, acquired an acid strength of $\frac{1}{5}$ per cent. oxalic acid; 100 cc. of the citrate of ammonia solution treated for an hour in the same way acquired an acid strength of 1.2 per cent. citric acid. The energy with which these acid solutions attack the undecomposed rock present in a superphosphate is shown in Series V. It is on account of these facts that Fresenius uses a neutral solution of the citrate salt and adopts the low temperature of 35°C., and Luck* in a second paper dwells with great emphasis upon this point in answer to the criticisms of a French chemist. But as the higher

*Zeitschrift fur anal. Chem. 14.

temperature is rendered indispensable on account of the presence of phosphates of iron and alumina in the great majority of superphosphates, this loss of ammonia and consequent acidity was prevented by digestion in a flask closed with a rubber cork.

The severest criticism upon the determination of reverted phosphates as an anaiytical process is the fact that when different quantities of substance are taken for analysis, different results are obtained. This most exceptional experience in chemical analysis, which is without a parallel in analytical determinations, requires a most careful examination, as this has done more than all else to cast discredit upon this analytical process. And yet it seems clear that this trouble can arise from only three sources :

(1.) Either the fertilizer under treatment has so large an amount of reverted phosphate present that when a large amount is taken for analysis the citrate is unable to take it all up, but is able to do so when a smaller amount is employed, or—

(2.) The citrate is an imperfect solvent for some form of reverted phosphate that may be present, either on account of the character of the latter, or on account of the method employed failing to dissolve it completely, the error showing more clearly as large quantities are taken for the analysis, or—

(3.) The discrepancy arises from the solvent action of the citrate upon the undecomposed rock present, a larger percentage of this being dissolved when smaller quantities are taken (*vide* Series I. and II.) The instances given by German experimenters, in the case of fertilizers containing 25 per cent. or more of phosphoric acid present as precipitated tricalcic phosphate, may be due mainly to the first of these causes. In such cases a smaller quantity must be taken, or it may be found that the higher temperature here recommended will so increase the solvent power of the citrate solution as to render this unnecessary. Whenever the phosphates of iron or alumina are present, it is evident from the experiments given that this discrepancy may be largely due to the second cause here assigned, namely, the method of the analysis, employing as it does the low temperature of 35° C. In order to ascertain whether the higher temperature of 70° C. to 40° C. will obviate the error from this second cause, 1, 2, 3, 4, and 5 grams of the above acid Navassa were treated each with 100 cc. of the citrate solution at this temperature, after the thorough washing out of the soluble phosphate.

SERIES XIII.

	P_2O_5 dissolved.	P_2O_5 undissolved.
Employing 1 gram substance	10.06 per cent.	1.94 per cent.
" 2 "	9.85 "	2.15 "
" 3 "	9.65 "	2.35 "
" 4 "	9.43 "	2.57 "
" 5 "	9.02 "	2.98 "

The very slight and uniform decrease of reverted phosphoric acid, about 0.20 per cent. for each additional gram taken, up to the 5 grams when the increase is a trifle larger, would certainly seem to show that every source of discrepancy but the third mentioned above had been obviated. When it is observed that in the last experiment 0.451 gram of reverted phosphoric acid is dissolved by the 100 cc. of citrate of ammonia solution at the temperature of 70° C. to 40° C., it certainly seems that nothing further could be desired as regards vigor of action upon the reverted forms. No other solvent suggested will approach the citrate solution in this important respect, when employed at the temperature here adopted.

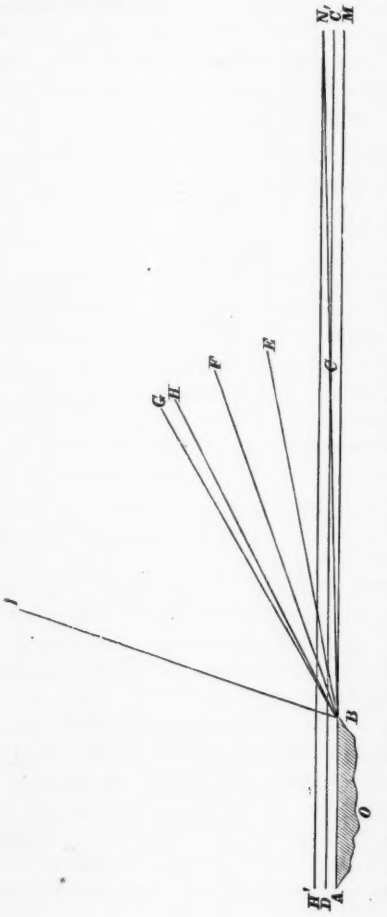
But as regards the third cause of discrepancy it is very different. This cause lies in the very nature of things and cannot be overcome. No reagent can be found that will dissolve the strictly reverted phosphates and not affect the undecomposed rock present. Nor is it desirable that any such reagent should be found, nor would it be just to employ it in case it were possible to find it. The whole idea upon which the determination of reverted phosphates rests is that the solvent action of the liquids of the soil is stronger than that of pure water, and is closely imitated by the solvent action of the organic salts of ammonia. Now there is a certain percentage of the raw phosphates that will always be taken up by this increased solvent power, and this percentage is practically and with justice considered equal in value to and reported as reverted phosphate.

The only difficulty that now presents itself is to so fix the amount of substance taken for analysis that the solvent action of the citrate solution upon the undecomposed rock shall dissolve such a percentage of this as shall when reported as reverted phosphate be a fair equivalent of its agricultural value. This conception here stated will be best illustrated by the accompanying diagram. 2 grams of the five forms of raw phosphates were treated with 100 cc. of citrate of ammonia solution at a falling temperature 70° C. to 40° C. for one hour, also two grams of the reverted phosphates

of iron and alumina containing 35.62 per cent. of phosphoric acid. The latter was dissolved to a clear solution.

SERIES XIV.

Apatite,	Phosphoric acid dissolved,		0.60 per cent.
South Carolina rock,	"	"	2.62 "
Bone Ash,	"	"	3.15 "
Navassa,	"	"	3.75 "
Curacao,	"	"	7.50 "
Reverted phos. of iron and alumina,	"	"	35.62 "



In the diagram use is again made of the figure of speech previously employed. AOB is the river bed, representing the phosphates soluble in water, completely covered by the line AM, the height of which represents the solvent power of water. The line BC represents the flats or the reverted phosphates lying just above the water, and slightly rising, since the different forms possess different degrees of solubility. The line DC, DC′ represents the solvent power of 100 c.c. citrate of ammonia solution at the temperature 70° C. to 40° C. The lines BE, B F, BH, BG and BI represent the banks rising at different angles on the different raw phosphates, Curacao, Navassa, Bone Ash, South Carolina rock, and Apatite here examined. These lines are all two inches in length, to represent the two

grams of substance taken. All of BC falls below DC or becomes soluble in the citrate solution, $\frac{1}{4}$ of BE, $\frac{1}{8}$ of BF $\frac{1}{11}$ of BH, $\frac{1}{10}$ of BG, and $\frac{1}{70}$ of BI, calculating from 30, 30, 36, 27, and 42 percentages of phosphoric acid present in the five samples.

Now suppose one gram of the above substances be taken to 100 cc. of citrate solution. This proportion is the same as 200 cc. citrate solution to the 2 grams of substance taken in the diagram. The line DC will be raised to H'N, twice as far above the surface of the water. Twice as large an amount of reverted phosphates will be taken up, viz. BN, and at the same time nearly twice as large an amount of the raw phosphates as shown in the figure. Thus repeating Series XIV., using however one gram of substance, there were dissolved as follows:

SERIES XV.

Apatite,	Phosphoric acid dissolved,		1.06 per cent.
Sou h Carolina rock,	"	"	
Bone Ash,	"	"	5.20 "
Navassa,	"	"	6.20 "
Curacao,	"	"	11.50 "

Attention is called to the nature of Curacao phosphate as brought out in this connection. Since by successive treatments with citrate of ammonia solution, or by employing a very small quantity for analysis, as $\frac{1}{70}$ of a gram, nearly all of this can be brought into solution, many chemists regard its phosphoric acid as nearly all in the available form, and an analysis made by one chemist on the identical sample used in this work reports the presence of 18 per cent. of reverted phosphoric acid. But the whole course of experiments given in this paper shows conclusively that it differs from the other raw phosphates only in degree of solubility, and is very far removed as regards this property from the true reverted forms, being, as shown in the diagram, based upon Series XIV., only $\frac{1}{5}$ as soluble at the best as the latter, and only about twice as soluble as Navassa rock and bone ash.

It seems almost certain from the agricultural experiments described in the last edition of Johnson's "Agricultural Chemistry," on the use of finely ground phosphates, that all the phosphoric acid in such phosphates becomes ultimately soluble in the soil. The division of phosphates therefore into the three forms, soluble, reverted, and insoluble, will have reference only to the time required to render them available for plant use. Supposing, in the

above diagram, that the solvent action of 100 c.c. of the citrate solution represents the solvent action of the liquids of the soil for a certain period of time, as *e.g.* one season, upon the forms of phosphate there represented, it is evident that all the soluble and reverted would be available the first season, that raw Curacao would all be rendered available in something over five seasons, the Navassa in eight seasons, and so on. The divisions, therefore, of soluble, reverted, and insoluble become indispensable as means of fixing commercial and agricultural values.

In conclusion, emphasis is laid upon the following points as brought out in this inquiry.

I. While the line of division between the reverted forms and the original insoluble forms of phosphoric acid may be sharply drawn in the case of an extremely insoluble phosphate, like apatite, in the case of the more easily soluble natural phosphates the two great classes overlap, one the other. Whatever solvent therefore is used for determining reverted phosphates the same will also dissolve a small percentage of the undecomposed phosphate that may be present; this percentage varying considerably according to the character of the insoluble phosphate. This percentage, representing as it does the comparative solubility which such insoluble phosphate possesses to the true reverted phosphates present, is justly considered as equivalent to an equal amount of the true reverted forms, and reported as such.

II. A slightly ammoniacal citrate of ammonia solution, alone of all the solvents that have been proposed, is a perfect solvent for all the forms of reverted phosphate, while at the same time not unduly dissolving the raw or insoluble phosphates present. Such a solution dissolves but a trace of the most difficultly soluble form of phosphate known, viz. apatite, and dissolves the other natural phosphates to a greater and greater extent as they approach in character the true reverted forms.

III. Neither the "Washington method" (method of Fresenius) nor the "Cincinnati method" can be relied upon to take up all the reverted phosphates that may be present.

IV. A modification of the method of Fresenius, consisting of the use of a higher temperature for the digestion, will meet all the requirements of the case, provided that the greatest care is taken to guard against acidity of the solvent and consequent excessive solution of the insoluble phosphates present. Whether the falling temperature of 70° C. to 40°C. as here explained, or a continuous tem-

perature of 65° C. for 30 minutes, be used, there will be but slight if any differencee in the results, provided that the solution of citrate be slightly ammoniacal, and the digestion be made in a closed flask. To the use of *acid* solutions of citrate, either acid at the beginning or the digestion or becoming acid during its continuance, are probably due more than to any other cause, the discrepancies in this analytical process.

XVIII. THE CONTAMINATION OF THE NEW YORK WATER SUPPLY.

BY DR ALBERT R. LEEDS

In this journal (vol. III, 98,) I have given under the title "Relative purity of city waters in the United States," the analyses of the drinking waters of many of our large cities, as performed by the same methods, and as made upon samples collected at or near the same date. As a necessary deduction from these results, the drinking waters arranged themselves in a series, in which the Brooklyn water occupied the place of honor as the purest, the Boston water the lowest place, being the most foul. The New York water, No. VI, fell in the middle of the series. The water supplies (judged by the samples analyzed) of Brooklyn, Rochester, Philadelphia, Baltimore and Washington, were pronounced satisfactory. Those of New York, Newark, Jersey City, Hoboken, Cincinnati, Oswego, Wilmington (Del.) and Boston, were stated to be contaminated. There was no question as to the contamination of the Newark (Passaic) water (No. 7), because its impurities were established by a multitude of analyses. And with regard to the Croton water the evidence of contamination was found in the facts:

1st. That its various impurities exceeded the limits which are allowable in a pure drinking water. 2nd. That its composition approached very closely to that of the Passaic, of whose contamination there could be no question; 3rd. That the water shed of the Croton contained sufficient sources of pollution to account for the figures obtained by analysis.

The results above summarized have been accepted without cavil, except in the case of the New York water supply. The pollution of the Boston water was so great that I could not credit my results without making a personal inspection of the sources of the Boston supply. This I did at a later season, and found not only were the first results more than confirmed but that the sources of pollution

were unmistakable. They were in the first place, the impurities thrown in by manufacturing communities ; and secondly, the enormous expansion of certain organic growths at favorable seasons of the year. The results relating to the water supply of Wilmington, Del., which were at first objected to by the uninformed local press of that city, were later on acknowledged by the health and city authorities to be warranted by the actual facts, and I am at present engaged on behalf of this city to investigate the precise nature and extent of the pollution.

Dr. E Waller in this journal (Vol. IV, p. 15), has objected to the New York water supply being characterized as contaminated, on the ground that his results, for a term of years, differ greatly from my own and are of a more favorable character. Also, for the reason that the Croton water shed cannot originate any sources of contamination ; and finally, that the health of New York is not, and has never been, such as to indicate the presence of any contamination in the water supply.

In reply, I would state :

1st. That the analyses of the New York water, on which Dr. Waller bases his opinion, have been incomplete, and data have been lost sight of, which are of the greatest value in forming a judgment as to the fact of contamination.

2nd. That certain of his methods are erroneous, and of necessity give a more favorable impression of the water than its true character warrants.

3rd. The analysis of the Croton water made June 23rd, 1881, does not stand alone, but is confirmatory of other previous analyses which led me to the conclusion that the Croton water was contaminated. It was not made, as Dr. Waller intimates, upon water turbid with sediment, but upon clear water drawn from the continuously flowing faucet in the Barclay street ferryhouse. I took it from this source as affording a very fair average sample of Croton water and I have no reason for supposing that it was otherwise.

4th. The condition of the Croton water shed is such as to account for the figures given in my analysis. The table of population given by Dr. Waller for the water sheds, does not explain the facts which exist ; for Rochester with but *half* the density of population on its Hemlock Lake water shed, that exists upon the Croton, has a far superior water supply, while Brooklyn on the other hand, with *double* the population, has a better supply than either.

5th. There are no data given, or so far as I know ever have been

given, upon which conclusions can be based connecting the vital statistics of New York with the character of its water supply. The face of Dr. Waller's assertion is not to be measured by its positiveness, but upon the appositeness and weight of the data upon which it is based, and of such data he has given us none.

It is extremely difficult, unfortunately, to connect the various diseases of a great community with the more or less deteriorated condition of its water supply. Unfortunately because if the connection were one easily proved, both chemist and medical practitioner could arrive with ease and certainty at results which are now open to more or less criticism. Whilst reiterating in various communications the statement that the water supply of Newark and Jersey City is contaminated, and in seasons of excessive drought like the summers of 1881 badly so, I have repeatedly challenged the physicians of these towns, to connect by irrefragible demonstration, the impurity of the water with any case of disease, and they have professed their inability so to do. I mention this, more particularly because the chemist is often accused by the physician of reporting a long series of decimals, without satisfactorily showing that any of the substances reported are specific causes of disease. Here, on the other hand, is a case where for years the water has been reported, on the strength of the oxidizable and oxidized nitrogenous matters which it contains, to be contaminated, and yet no physician has proven, or attempted to prove, that any particular case of disease has been due to drinking it. In the population of 50,000 people inhabiting the town of Paterson, which pours all its sewage into the Passaic at a point 16 miles above the intakes of the pumping stations of Newark and Jersey City, and in the city of Passaic with its considerable population likewise sewering into the Passaic river at a point only half this distance, there must be many cases of typhoid fever, yet no one has ever shown that a single case of typhoid fever has been originated in Newark and Jersey City by drinking the Passaic water. From what source then does Dr. Waller derive the statistics upon which he infers the purity of the Croton water from the death-rate of the New York population?

To return, however, to the more serious sources of error in the methods made use of by Dr. Waller.

1st. With reference to the incompleteness of his analyses. He has charged me with indefiniteness in the use of the terms "nitrites" and "nitrates." I fail entirely to see any indefiniteness. I used these terms because they were the proper generic names of the sub-

substances I determined. Nitrites and nitrates were the bodies meant, and the bodies whose determination *as such*, I regarded as of importance. The results were calculated to nitrites (HNO_2) and nitrates (HNO_3), and not to the "nitrogen in nitrites or nitrates," because I had no interest in knowing the amount of the nitrogen, could not have made use of it in my calculations, and think its estimation would have been absurd. If the organic carbon and nitrogen had been determined instead of the free and albuminoid ammonia, then indeed I should have calculated out the nitrogen in the nitrates, in order to compare this amount with the organic nitrogen, and thus to find the correct ratio between the organic carbon and nitrogen in the organic compounds present.

There is no evidence that Dr. Waller had estimated the nitrates until the summer of 1881, nor does he appear to have remarked the fact that the Croton water at times gives the reaction for *nitrous acid*. The latter I regard as a most important fact (although too frequently overlooked), because it is very rare that a decided reaction for nitrites is obtained in drinking-waters, and only when the oxidizable nitrogenous compounds are caught in the very transient stage of nitrites, and before their oxidation to the final condition of nitrates. If in *river-waters* the reverse transformation is effected, and any nitrites undergo reduction to nitrites, I have not yet in my own experience encountered a case in which there was any reason for supposing that such a reverse transformation has been effected.

An unusual opportunity for noting this oxidation of albuminoids into nitrites and finally into nitrates, is afforded when the pure mountain-stream waters of the Passaic, after leaping the Great Falls, come straightway into contact with the sewage of the population of 500,000 inhabitants of Paterson. The albuminoid ammonia increased *per saltum* (Sept. 6th, 1881) from 0.014 parts per 100,000, to 0.225 parts. It then fell away in the course of 2 miles flow to 0.133 parts, and at the end of 7 miles flow had fallen to 0.0215 parts per 100,000. The nitrates at the corresponding points were 0.185 parts; 2.183; 0.722; 1.388 parts. The nitrites at the same points were none; a considerable amount the quantitative determination of which was lost); a trace; none.

It is seldom, however, that the reaction for nitrites is obtained in the Passaic water after it has flowed 16 miles, and has been delivered by the pumping-stations to the consumers in Newark and Jersey City. The oxidation of the albuminoids during its long flow is so complete, that while the amount of nitrates is always

high, the amount of nitrites is usually quite inappreciable. And the same is generally true of river-waters. In the 23 samples of water, the composition of which is given in the table upon the "Comparative Purity of City Waters in the United States," no reaction for nitrites happened to be obtained in the case of any one of them. And yet the Boston water required the enormous amount of 1.77 parts to the 100,000 of oxygen to effect the oxidation of its organic matters, and contained 1.24 parts of nitrates. Sometimes the Passaic water does afford the nitrous reaction, but in these cases I have attributed it to the non-oxidized sewage of Newark which has only to flow upward for a couple of miles before reaching the intakes of the pumping-stations, rather than to the sewage of Paterson, which has to flow downward 16 miles before reaching the same points, and is usually completely oxidized in its long exposure.

The fact that at times I have obtained from the Croton the nitrous reaction, in one instance a reaction strong enough to admit of quantitative estimation, has had due weight in forming my estimation of its purity.

2nd. The method which Dr. Waller employed of determining the amount of oxygen required to oxidize the organic matter is erroneous, and of necessity gives a more favorable impression of the water than its true character deserves. In making this statement, I am aware of what has been written in favor of this method, but see no reason for regarding it otherwise than erroneous. If it is desired to oxidize the organic matter in drinking waters by potassium permanganate, so as to destroy the possibly noxious substances, the water to which the potassium permanganate has been added is boiled before it is thought entirely safe to drink. If the potassium permanganate is to be standardized by an organic substance, as it is in this instance by oxalic acid, the standardization is performed by heating the solution of oxalic acid or of permanganate. But in making the determination of the organic matter in the water itself, this method proposes it should be done at a variable common temperature. Of course, only a small amount of the organic matter in the water is thus oxidized, and although it is claimed that this part is the really dangerous putrescible portion, the fact still remains that only a part of the organic matter is attacked by the permanganate in the cold, and the method is thus far erroneous. Still more, the method lacks the essentials of an accurate volumetric estimation, and does not admit of arriving at a certain end reaction. I tried the method, and after satisfying myself of its essential

inaccuracy, adopted what appeared to me the most satisfactory and definite process, that of titration at the boiling point.

Dr. Waller's objections to this method are fanciful rather than real. The impurities in the permanganate and sulphuric acid, though actually present, are too small to admit of any appreciable influence on the results. The objection that the chlorides in the water would affect the results has greater weight, but in practice these errors, like those produced in Tidy's modification of the permanganate process by the presence of chlorides, are too small to be considered. I am fortunately able to bring an experiment, performed on polluted natural waters themselves, to confirm this statement. The Passaic water (Sept. 6th, 1881), at the intake of the Jersey City Reservoir, contained in 100,000 parts:

	LOW TIDE.			HIGH TIDE.		
	E. Bank.	Middle.	W. Bank.	E. Bank.	Middle.	W. Bank.
Chlorine,	5.73	2.10	3.40	55.88	44.75	59.75
Oxygen required	0.55	0.44	0.44	0.47	0.60	0.76

The differences had been caused by the backing up of the river waters, on meeting the tidal wave from the sea.

On the same day, and upon the same two tides, the differences for the corresponding points at the Newark Reservoir, three-quarters of a mile further up stream, were :

	LOW TIDE.			HIGH TIDE.		
	E. Bank.	Middle.	W. Bank.	E. Bank.	Middle.	W. Bank.
Chlorine,	2.185	1.17	2.505	21.13	25.00	14.50
Oxy. required,	0.62	0.56	0.43	1.80	2.82	2.82

But it may be objected to these results, that the oxydizable organic matters in the various samples varied greatly, so that no inference could be drawn with regard to the influence of the chlorine itself.

This objection does not apply to the water taken at intervals of about a mile down the stream, upon the same day (Sept. 6th, '81).

Beginning at a point below the Tail Race at Passaic Mills, we found for samples :

No.	75	76	77	78	79	80	81	82	83	84
Chlorine,	0.585	0.60	0.94	0.57	1.57	1.005	0.90	0.56	1.10	2.505
Oxy. required	0.53	0.50	0.49	0.52	0.51	0.59	0.47	0.47	0.47	0.43

It may be objected, however, that all these results merely raise a strong presumption in favor of the proposition that the small

amounts of salt in river water do not appreciably raise the figures obtained by permanganate at the boiling point. They do not prove it ; for proof a suitable experiment is demanded.

In the first place, it was necessary to determine the coefficient of change, when potassium permanganate is boiled with *distilled* water. Both in the cold and on heating, potassium permanganate changes in acidulated solution with distilled water, and the amount of this change has to be determined in both cases, and a corresponding deduction has to be made from the amount obtained with an equal volume of the natural water under examination, I thus obtained :

100 cc. distilled water, Miller's method (3 hr.) 0.9 cc. permanganate.
100 cc. distilled water, Kubel's method, – — 0.9 cc. permanganate.

Another test by Kubel's method on a different sample of distilled water, and with different permanganate solution, gave 1.15 cc. permanganate.

In other words, in order to make use of Miller's method with any expectation of arriving at accurate results, it is necessary in each case to apply a correction due to the alteration of the permanganate alone. This and many other precautions C. M. Tidy (Jour. London Chem. Soc., xxxv., p. 46) has taken, in order if possible to bring Miller's process into a form capable of yielding regular and accurate results. But I do not find in Dr. Waller's account of Miller's method, as he employed it, that any such precautions were taken. The influence of these corrections may be perceived by considering the great changes thereby produced in Dr. Waller's results. For instance, for the first five months of the year 1879, he obtained 0.072 parts of oxygen per 100,000, as the amount required to oxydize the organic matter. This would be equal to 0.72 cc. permanganate solution used. If, as is possible, he had obtained 0.9 cc. as the correction to be applied by the use of the permanganate solution with *distilled water alone*, there would have been left a minus quantity, or 0.018 parts of oxygen, as the amount required to oxydize the organic matters in the Croton water.

Dr. Tidy has endeavored to find the extent to which the chlorides affect the accuracy of the modified Miller's method, and has found that their influence, even when present in very large quantities, practically *nil*. A similar remark applies to the effect of chlorides on the results afforded by Kubel's method. For instance, in an experiment in which 100 cc. of *distilled water* decolorized on boiling 1.15 cc. permanganate, it decolorized only 1.3 cc. permanganate

after sodium chloride had been added in amount corresponding to 400 parts in 100,000. This amount vastly exceeds any quantity likely to exist in river or well waters.

In practice, however, I think it is better, whenever the amount of sodium chloride is large, to add the percentage indicated by the chlorine determination to the distilled water used in making the blank experiment. If the results thus obtained differ from those obtained by the use of the 100 cc. distilled water alone, they can be deducted, instead of the latter, from the number of cubic centimeters of permanganate obtained with the natural water.

3d, and finally. The analysis of the Croton water made June 3d, 1881, does not stand alone, but is confirmatory of other previous analyses, which led me to the conclusion that the Croton water was contaminated.

The best proof of this statement is to be found in an analysis made only three days before the one intended for the general comparison with the drinking water of all the other large cities, or on June 20th, 1881. It was made upon unfiltered but perfectly clear water, drawn from the same place, the constantly running faucet in the Barclay street ferry-house. It shows by its close agreement with the results obtained on the sample taken June 23d that neither analyses gave high figures by accident, but both are to be regarded as mutually confirmatory results of the actual average condition of Croton water at that time.

	CROTON WATER, June 20th, 1881.	PARTS PER 100,000. June 23d, 1881.
Free ammonia	0.004	0.0027
Albuminoid ammonia	0.027	0.0270
Oxygen required	0.73	0.810
Nitrites	None.	None.
Nitrates	0.6105	0.8325
Chlorine	0.26	0.35
Total hardness	3.00	3.30
Permanent hardness	2.60	——
Temporary hardness	0.40	——
Total solids	9.00	11.80
Mineral matters	2.50	5.00
Organic and volatile matters	6.50	6.80

XIX.—ON THE ESTIMATION OF PHOSPHORIC ACID AS MAGNESIC PYROPHOSPHATE.

BY THOMAS S. GLADDING.

In a valuable paper on the determination of phosphoric acid as magnesic pyrophosphate (*Amer. Chem. Journal*, vol. 1, 391) Gooch has demonstrated that this method, far from being as accurate as is generally supposed, is peculiarly liable to sources of error, which cause a considerable overestimation of the phosphoric acid actually present.

The great importance of this analytical determination from a commercial point of view, gives enhanced interest to any inquiry as to its accurate execution. In view of the vast capital at present engaged in the conversion of phosphatic materials into artificial fertilizers, and in view of the fact that chemists frequently differ so widely in this important determination, the necessity, both for the interests of commerce and the credit of science, of an accurate and rigid method can be clearly understood. In the case of large and valuable cargoes sold upon chemical analysis, the slight error of even $\frac{1}{10}$ of a per cent. in the estimation of phosphoric acid causes a difference in value ranging from twenty-five up to two hundred dollars.

I have been led by these considerations to a careful examination of the modes of procedure at present recommended by the best authorities and which are in common use among chemists.

In the latest American edition of *Fresenius' Quant. Analysis* the directions are, to add to the neutral or moderately ammonical solution of the phosphate, magnesia mixture in slight excess, and after letting stand for some time, $\frac{1}{3}$ its volume of strong ammonia solution. In the case of a previous precipitation as phospho-molybdate of ammonia, this precipitate is dissolved in weak ammonia and the neutral or slightly ammoniacal solution, treated as above.

The method recommended by the convention of chemists of the different experimental stations of Germany (held in Dec. 1881) is translated as follows: (Zeit. fur. Anal. Chem. 21, p. 290) "The beaker glass is now placed under the funnel, the filter pierced with a platinum wire, the precipitate washed into the beaker glass with $2\frac{1}{2}$ per cent. ammonia liquor, and dissolved by stirring with a glass rod, and then enough of the $2\frac{1}{2}$ per cent. ammonia liquor added, to bring the volume of the liquid up to about 75 cc. To 0.1 gram phosphoric acid, 10 cc. magnesia mixture are now dropped in ac-

companied by a constant stirring up." In a foot note is added "a gradual addition of the magnesia mixture is advisable under all circumstances even when the ammoniacal solution of the molybdate precipitate is previously nearly neutralized by addition of hydrochloric acid." This mode of procedure differs from that given in "Fresenius" principally in the one point of *insisting on a g adual* addition of the magnesia mixture. In both methods a strongly ammoniacal magnesia mixture is employed and this is added to a more or less strongly ammoniacal solution of the phosphate.

On the other hand the method giving the best results in the hands of Gooch, consisted in the addition of a *neutral* magnesia mixture to a *neutral* solution of the phosphate or still better, especially in the case of a previous precipitation as molybdic salt, in a double precipitation of the ammonia-magnesia-phosphate.

In testing the above methods great care was exercised in the preparation of the standard solutions. These were made by the use of chemically pure microcosmic salt. Upon careful ignition of this salt in a covered platinum capsule 5.1415 grams gave 2.5110 grams of phosphate of soda as residue or 34.00 per cent. of phosphoric acid against 33.98 per cent. required by theory. With this salt, two solutions were prepared, the one containing 4.121 grams of the salt in a liter or 0.035 grams $P^2 O^5$ in every 25 cc., the other 20.605 grams to a liter or 0.175 grams of $P_2 O^5$ in every 25 cc. 25 cc. were measured off with great accuracy by means of a pipette holding exactly 25 cc. by careful weight test, when filled to the mark. This was then carefully rinsed out. The small neck of the pipette, makes it easy to measure a liquid to within $\frac{1}{100}$ cc. by this method. In addition to the experiments made with these solutions, others were made by direct weighings of the microcosmic salt.

Method I.

To the solution of the phosphate, diluted to 50 cc. and made slightly *alkaline* by the addition of $\frac{1}{2}$ cc. of ammonia liquor, the regular ammoniacal magnesium chloride mixture was added, 4 cc. to every 25 cc. of solution I. and 18 cc. to every 25 cc. of solution II. After letting stand for 15 minutes, 25 cc. of strong ammonia solution were added, and the liquid filtered off after about 30 minutes. The washing was performed with strong ammonia solution and was continued until 10 drops of the washings gave no precipitate with acid of silver nitrate when acidified. In these and all subsequent experiments the filter papers were each weighed, that

an accurate weight allowance might be made for filter ash, and the precipitate was strongly ignited over a blast lamp for several minutes, until white. Two series of experiments were made, the one without a previous precipitation as phospho-molybdate of ammonia, the other with such previous precipitation.

A. Without previous precip. as molybdate salt.

Amount taken.	$Mg_2P_2O_7$ found.	P_2O_5 found.	P_2O_5 found.	Error.
1 25 cc. sol. I.	0.0559	0.0350	0.03575	2.14 p. c.
2 "	0.0557	"	0.03563	1.80 "
3 "	0.0559	"	0.03575	2.14 "
4 "	0.0557	"	0.03563	1.80 "
5 25 cc. sol. II.	0.2770	0.1750	0.17718	1.25 "
6 "	0.2763	"	0.17673	0.99 "
7 "	0.2760	"	0.17654	0.88 "
8 "	0.2762	"	0.17666	1.95 "

Average error 1.50 per cent.

B. With previous precip. as molyb. salt.

Amount taken.	$Mg_2P_2O_7$ found.	P_2O_5 required.	P_2O_5 found.	Error.
1 25 cc. sol. I.	0.0559	0.0350	0.03575	2.14 p. c.
2 "	0.0559	"	0.03575	2.14 "
3 "	0.0558	"	0.03569	1.97 "
4 "	0.0556	"	0.03555	1.57 "
5 25 cc. sol II.	0.2768	0.1750	0.17705	1.17 "
6 "	0.2780	"	0.17782	1.61 "
7 "	0.2774	"	0.17744	1.40 "
8 "	0.2776	"	0.17756	1.46 "

Average error 1.68 per cent.

METHOD II.

The same course was followed as in method I, excepting that the solution of phosphate was exactly NEUTRALIZED before the addition of the magnesia mixture.

A. Without previous precip. as molyb. salt.

Amount taken.	$Mg_2P_2O_7$ found.	P_2O_5 required.	P_2O_5 found.	Error.
1 25 cc. sol. I.	0.05570	0.0350	0.03563	1.80 p. c.
2 "	0.05570	0.0350	0.03563	1.80 "
3 25 cc. sol. II.	0.2758	0.1750	0.17641	0.81 "
4 "	0.2757	0.1750	0.17635	0.77 "
5 "	0.2753	0.1750	0.17609	0.62 "

Average error 1.16 per cent.

B. With previous precip. as molyb. salt.

Amount taken.	$Mg_2P_2O_7$ found.	P_2O_5 required.	P_2O_5 found.	Error.
1 25 cc. sol. I.	0.0556	0.0350	0.03555	1.57 p. c.
2 "	0.0556	0.0350	0.03555	1.57 "
3 25 cc. sol. II.	0.2770	0.1750	0.17718	1.25 "
4 "	0.2772	0.1750	0.17731	1.32 "

Average error 1.43 per cent.

Method III.

To the solution of phosphate, 75 cc. in volume and quite strongly ammoniacal, the magnesia mixture was added at the rate of one drop a second, the contents of the beaker being kept in constant rotation. The magnesia mixture was added from a burette, the rapidity of flow being regulated by a glass cock, or still better from an ordinary Mohr burette, the flow being regulated by a screw-clamp. The beaker was slightly inclined, so as to receive the drops on its side. The magnesia mixture by this arrangement flowed down the side of the beaker in a thin stream and an exceedingly gradual admixture with the phosphate solution was thereby secured. After the addition of the magnesia mixture, 25 cc. of strong ammonia were added and the whole allowed to stand for two or three hours. Very strong ammonia water was used in the washing as in several cases a tendency to run through the filter paper was observed, when a weaker ammonia solution happened to be employed. One part conc. ammonia water to three parts water was employed.

A. Without previous precip. as molybdate salt.

	Am't. taken.	$Mg_2P_2O_7$ found,	P_2O_5 required,	P_2O_5 found,	Error.
1.	25 cc. sol. I.	0.0547 g.	0.0350 g.	0.03499 g.	0.00 p.c.
2.	"	0.0547	0.0350	0.03499	0.00 p.c.
3.	25 cc. sol. II.	0.2736	0.1750	0.17500	0.00 p.c.
4.	"	0.2735	0.1750	0.17494	0.03 p.c.
5.	"	0.2738	0.1750	0.17513	0.07 p.c.
6.	0.5231 g. micro. salt	0.2775	0.17775	0.17750	—0.14 p.c.
7.	0.5120	0.2717	0.17397	0.17379	—0.10 p.c.

Average error. 0.05 p. c.

B. With previous precip. as molybdate salt.

	Am't. taken.	$Mg_2P_2O_7$ found,	P_2O_5 required,	P_2O_5 found,	Error.
1.	25 cc. sol. I.	0.0548 g.	0.0350 g.	0.03505	0.14 p.c.
2.	"	0.0548	0.0350	0.03505	0.14 p.c.
3.	25 cc. sol. II.	0.2736	0.1750	0.17500	0.00 p.c.
4.	"	0.2734	0.1750	0.17488	—0.07 p.c.
5.	"	0.2732	0.1750	0.17475	—0.14 p.c.
6.	"	0.2733	0.1750	0.17481	—0.11 p.c.

Average error. 0.10 c.

METHOD IV.

To the solution containing the phosphate, either neutral or ammoniacal, the magnesia mixture was added, and then 25 cc. of conc. ammonia water. After letting stand for thirty minutes, the liquid was filtered off and the precipitate thoroughly washed on the paper. The precipitate was then dissolved on the paper with dilute HCl, and washed into the original beaker. 25 cc. of conc. ammonia solution were now added to the filtrate, no care being taken to add it gradually. After the flocculent precipitate had settled, 5 cc. magnesia mixture were added and the whole allowed to stand several hours. The second precipitation was found to be incomplete without the addition of this extra magnesia mixture.

A. Without previous precip. as molybdate salt.

	Am't. taken.	$Mg_2P_2O_7$ found,	P_2O_5 required.	P_2O_5 found.	Error.
1.	25 cc. sol. II.	0.2736 g.	0.1750 g.	0.1750 g.	0.00 p.c.
2.	"	0.2735	0.1750	0.17494	0.03 p.c.
3.	"	0.2736	0.1750	0.17500	0.00 p.c.
4.	0.5095 g. micro salt.	0.2712	0.17313	0.17336	0.13 p.c.
5.	0.5703 "	0.3030	0.19379	0.19380	0.00 p.c.
6.	1.0108 "	0.5372	0.34347	0.34361	0.04 p.c.

Average error. 0.03 p. c.

B. With previous precip. as molybdate salt.

	Am't. taken.	$Mg_2P_2O_7$ found.	P_2O_5 required.	P_2O_5 found,	Error.
1.	25 cc. sol. II.	0.2738 g.	0.1750 g.	0.17512 g.	0.00 p.c.
2.	"	0.2736 g.	0.1750 g.	0.1750 g.	0.07 p.c.

Average error. 0.035 p. c.

Still another method was investigated as follows: To the *neutral* solution of phosphate was added a *neutral* magnesia mixture in slight excess. No precipitate appeared except in the case of very conc. solutions. An extremely weak ammonia water was then added, drop by drop, to the liquid, to which was imparted at the same time a constant rotary motion. An exceedingly granular, pure precipitate was thus produced. The results obtained were perfectly accurate, but as the precipitate was very light and apt to fly, when removed from the paper into the capsule, for ignition, and as the method involved the extra labor over Method III, of first neutralizing the solution, it was not carried out in full.

A series of determinations by these several methods, on different fertilizers received in the course of business, was also carried out. The average error is calculated from the results obtained by Method IV, regarded as correct.

Fertilizer	Method I. P_2O_5 p. c.	Method II. P_2O_5 p. c.	Method III. P_2O_5 p. c.	Method IV. P_2O_5 p. c.
Superphosphate	10.88			10.50
"		9.49	9.38	9.37
Acid phosphate	14.65			14.20
"	15.20			14.75
Tankage	16.96			16.06
"		16.86		16.44
Bonemeal	22.20			21.60
"		21.83		21.51
Rock phosphate	25.15	24.96		24.45
"		28.93	28.32	28.39
Bone-ash	30.25		29.76	29 80
Bone-black			34.39	34.33
	Av. error 0.56 p. c.	Av. error 0.36 p. c.	Av. error 0.04 p. c.	

A study of these results demonstrates that the common method of precipitation as carried out under Methods I and II will invariably give results too high. In fertilizer work the error thus introduced will vary all the way from one or two-tenths of a per cent. when the solution is carefully neutralized and when it chances to be very dilute, up to six-tenths per cent. or even higher when the solution is more concentrated, or when molybdate of ammonia is present, or where the solution is strongly or even moderately ammoniacal when the magnesia mixture is added. This great variation of error undoubtedly explains many of the differences that have occurred in

the analyses of different chemists. The errors thus obtained clearly arise from the too sudden formation of the ammonia-magnesia phosphate precipitate, the precipitate carrying down with it extraneous matter, the error increasing, of course, when a large amount of molybdate of ammonia is present. When no foreign matter is present and the exact theoretical amount of magnesium chloride is contained in the solution, this sudden precipitation is not injurious, as is seen in Method IV. Method III when carried out as here described gives accurate results even in the presence of molybdate of ammonia. This method, as it does not necessitate any previous neutralization of the solution with hydrochloric acid, and requires but one precipitation is the one to be most highly recommended. Strong ammonia water should be added in liberal amount to the solution, and also employed in the washing.

The breaking of the filter paper and washing of the molybdate salt into the beaker, without filtering the solution cannot be recommended. However thoroughly the yellow salt may be washed, there is always a slight residue insoluble in ammonia water. A solution and filtration of the molybdic precipitate is therefore necessary in accurate work.

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ABSTRACTS.

Abstracts from *Journal of the Chemical Society*, London, by Arthur H. Elliott, Ph. B., F.C.S.

On the Volumetric Estimation of Bismuth in the Form of Oxalate. BY M. M. PATTISON MUIR, M. A., F. R. S. E. AND C. E. ROBBS, B. A. B. Sc. (Vol. XL., p. 1.)—Authors describe a process which depends upon the fact that potassium oxalate precipitates Bismuth as $Bi_2(C_2O_4)_3K_2C_2O_4$. The method of procedure is as follows:—

A solution of the bismuth in the minimum quantity of nitric acid is evaporated till only enough acid remains to give a solution when hot; a large excess of glacial acetic acid is added and the whole transferred to a measuring flask, washing with acetic acid (1:1). Standard potassium oxalate is now added till there is twice as much of this salt present as there is bismuth oxide; the flask is filled to the mark with acetic acid (1:1) and after shaking and allowing to

settle, a portion of the fluid is filtered through a dry filter and the excess of potassium oxalate determined by a standard solution of permanganate.

The bismuth must first be separated from other heavy metals preferably as oxychloride. The double bismuth and potassium oxalate is soluble in potassium oxalate; but precipitates with excess of strong acetic acid.

Action of Water on Bismuth Iodide. A Lecture Experiment. By M. M. Pattison Muir. (Vol. XL., p. 4.)—This experiment illustrates the influence of time, temperature, and mass, upon chemical change. Author pours solution of BiI_3 in hydriodic acid, into cold water, water at 90°—100° C. and large quantity of cold water. In the first brown Bi I_3 is formed, in the second red BiOI, and small quantity of red BiOI in the third.

Aluminium Alcohols. Part II. Their Products of Decomposition by Heat. By J. H. Gladstone, Ph. D., F. R. S., and Alfred Tribe, F. C. S. (Vol. XL., p. 5.)—This paper is a continuation of the highly interesting experiments of the Authors upon aluminium alcohols. The authors state that the general result of heating these alcohols is the formation of two sets of products,alumina and the alcohol and olefine, or alumina and the ether. They describe the products obtained by heating 1, aluminium phenylate, 2, aluminium para-cresylate, 3, aluminium thymolate; 4; aluminium beta-naphthylate, 5, aluminium gamma naphthylate. Among the products mentioned we note phenyl ether, cresyl-ether, metacresyl-ether, beta-naphthyl-ether. The paper is full of interesting facts and this abstract gives but a faint idea of the ground covered by the investigations.

On the Action of Oxides on Salts. Part IV. Potassic Chlorate and Ferric Oxide. By Edmund J. Mills, D. Sc., F. R. S., and George Donald. (Vol. XL., p. 18.)—Experiments of authors go to prove that practically 3 Fe_2O_3 will expel O_{10}. And in conclusion they state "that the case of chemical change which we have under consideration presents nothing abnormal or peculiar in its features. From the carbonate, an oxide of carbon is the matter expelled; from the chlorate, an oxide of oxygen." The name *catalysis* ceases to have any reason for its existence.

In a supplementary note it is stated that the action of mangan-

ese dioxide upon potassic chlorate is the same as that of an oxide on a salt.

Gamma and Beta-Amylan; Constituents of Some Cereals. By C. O'Sullivan. (Vol. XL., p. 24.) — Author describes two bodies of the same percentage composition as starch, one insoluble in water, and the other soluble. Barley contains 2 per cent of gamma-amylan,and not more than 0.3 per cent. of beta-amylan. Wheat and rye contain 2.25 per cent. of beta-amylan, and not over 0.1—0.05 per cent. of gamma-amylan.

Note on the Action of Ethyl-Chlorocarbonate on Benzene in presence of Aluminum Chloride. By Edward H. Rennie, M. A., B. Sc. (Vol. XL., p. 33.)—Author states that ethyl-chlorocarbonate acts upon benzene in presence of alumina chloride in the cold producing carbonic anhydride, hydrochloric acid and *ethyl benzene*. Also states that ethyl-chlorocarbonate, by contact with aluminium chloride in the cold, yields carbonic anhydride and mono-chlorethene. When ethyl-chloroacetate is substituted for ethyl-chlorocarbonate, no reaction takes place.

On Benzyl-Phenol and its Derivatives. By Edward H. Rennie, M. A., B. Sc. (Vol. XL., p. 33.) — Author describes the preparation of the monosulphonic acid, potassium nitrobenzyl-phenol-sulphonate, potassium bromo-benzyl-phenol-sulphonate, and trinitro benzyl-phenol, and gives descriptions of their properties. Author appears to think that benzyl-phenol is a para derivative.

On the Steeping of Barley. By Edmund J. Mills, D. Sc., F.R. S., and J. Pettigrew. (Vol. XL., p. 38.) This paper treats of the action of water, containing different quantities of calcium carbonate and calcium sulphate in solution, upon barley. In the case of calcium carbonate, as the latter diminishes the extract decreases, and the nitrogenous matters in the extract increase; but distilled water extracts less of the nitrogen compounds than 0.0224 per cent. solution of calcium carbonate. With calcic sulphate the total extract or nitrogen per unit of calcic salts is much greater than with the carbonate. The paper also contains an analysis of Burton water, and its effect upon the steeping of barley. It is shown that the extract decreases as the water is diluted with distilled water. Factitious waters were made containing some of the important constituents of the Burton water; and as a result of

steeping barley it was found that with calcic sulphate and sodic chloride, more extract and less nitrogenous matter was obtained than with Burton water; while with calcic carbonate and sodic chloride the extract is about the same and the nitrogen less than with Burton water. The general effect of a calcic solution is to keep back the nitrogen within the grain. "The special esteem in which the Burton water is held may therefore be due to its nitrate, which is well known to have a highly stimulant action in the germinating of malt—a process which demands much oxygen."

Researches on the Relation of the Molecular Stricture of Carbon Compounds to their Absorption Spectra. By W. N. Hartley, F. R. S. E. (Vol. XL., p. 45.) Part VI.—*On the constitution of Pyridine, Picoline, Quinoline, and Cyanuric Acid.*

After experimenting on Hydrocyanic Acid, the author comes to the conclusion that: "The simple union of carbon to nitrogen does not cause selective absorbtion of the ultra violet rays." A number of diagrams from photographs of absorption spectra are given and comparison is made between the different spectra. Author thinks it probable that in benzene each carbon atom is united with three other carbons. A full idea of the paper cannot be given without the diagrams.

On Peppermint Camphor (Menthol) and some of its Derivatives. By R. W. Atkinson, B. Sc., and H. Yoshida. (Vol. XL., p. 49.) Authors describe Menthone $C_{10}H_{18}O$, give its vapor density as 77.45 and 76.69; it boils at 206.3° C, and its specific rotary power is [alpha] j = + 21.16°. Its specific gravity at different temperatures as well as its rate of expansion and molecular refraction are also given. Menthone $C_{10}H_{18}$ is also described; it boils at 167.4° C, its specific gravity power is [alpha] j = + 13.25°. The specific gravity, rate of expansion and molecular refraction are also given.

On the Composition of Pennant Grits in Contact with and at a Distance from Carbonaceous Deposits. By Edward Wethered, F. C. S., F. G. S. (Vol. XL.,) A comparison of a number of analyses of the grits that accompany coal seams. The author finds that these become more argillaceous when in contact with the coal, and assigns the cause to the carbonic acid given off from the decomposing vegetable matter, acting upon the freshly formed inorganic sediment; decomposing all silicates except silicate of alumina.

XXX.—ON THE PROPERTIES OF PURE METALLIC ALUMINUM.

By J. W. Mallet.

In view of the marked effect upon the properties of the individual metals, which is often produced by the presence of even very small quantities of foreign substances, metallic or non-metallic, it is very desirable that when really pure specimens can be had, prepared and tested with unusual care, their properties shall be noted and recorded. Comparatively few of the metals have probably been procured and examined in a state of the highest attainable purity, and comparatively few chemists have learned by personal experience how very difficult it is to get rid of the last detectable traces of foreign matter. This difficulty is especially great in the case of those metals least easily reduced to the metallic state, among which may be counted aluminum.

The writer, having expended much time and labor upon obtaining pure metallic aluminum to be used in his work*, on the atomic weight of this element, succeeded in preparing enough for that purpose, and incidentally noted the following facts in regard to the metal—not forming a complete description of all its properties, physical and chemical—but yet deserving, perhaps, of being placed on record.

The crude (commercial) aluminum, used as the source of the pure metal, was found to contain:

Aluminum (by difference)	96.89
Iron	1.84
Copper	Trace
Silicon	1.27
	100.00

This was converted into bromide by treatment with liquid bromine, observing the precautions described in the paper (in Phil. Trans.) above referred to. A large quantity of the aluminum bromide—more than two kilogrammes altogether—was purified by careful fractional distillation, with due regulation of temperature, repeated a number of times, until the product was perfectly colorless, and on solution in water gave no evidence of the presence of

* Philosophical Transactions—Pt. III., 1880.

impurities, which were searchingly tested for. The reduction of the metal from this purified bromide was accomplished, but with much difficulty and great loss, by heating with pure sodium in a crucible made of, or lined with, a mixture of purified and dry alumina and sodium aluminate. The large globules of aluminum obtained were severally re-fused before the blowpipe on a support of alumina, and were then exposed for a short time to the action of hydrochloric acid on the surface, well washed with water, and dried. On solution of portions of the globules in pure hydrochloric acid, careful testing failed to show the presence of any of the impurities which might have been taken up from the materials and apparatus used. The entire absence of sodium was specially verified.

The color of this pure aluminum was perceptibly whiter than that of the commercial metal—on a cut surface very nearly pure tin-white, without bluish tinge, so far as could be judged from the small pieces examined. The lustre, too, was very much that of tin in a fresh, untarnished condition.

The metal was distinctly softer than before purification. Hence its fracture was not easily observed, but seemed to be very fine grained, with some appearance of fibrous silkiness. The malleability was undoubtedly improved, the metal yielding easily to the hammer, and bearing distortion well by flattening in two or three directions without cracking. It seemed to be sensibly less hardened by hammering than the ordinary metal of commerce.

The specific gravity was carefully determined at 4° C., and the mean of three closely concordant experimental results gave the number 2.583 as referred to water at the same temperature.

Hence the "atomic" volume=10.45, near that of gold (10.19, if the sp. gr. be taken as 19.295, the value found by Roberts for the large trial-plate of fine gold of the English mint), and standing to that of iron nearly as 3:2 (the at. vol. of the latter being 7.11, if the sp. gr. be taken=7.88, as found by Caron for pure iron fused in the oxy-hydrogen flame).

The specific heat was determined by means of Bunsen's ice calorimeter and found to be = .2253 as a mean for the range of temperature 0°—100° C., a number rather higher than those obtained by Regnault and Kopp. This number multiplied by 27.02, the atomic weight found in the experiments for which the metal was prepared, gives the product 6.09 as the atomic heat.

The quantity of pure material at command was not sufficient to

allow of any determination of the conductivity, though this would be an interesting point to examine, in view of the high conducting power of the commercial metal, and the probability that the value would be notably raised by the removal of impurities. An imperfect attempt was made to measure the expansion by change of temperature, using for the purpose determinations of specific gravity in water at widely different temperatures, and correcting for expansion of water and glass, but the quantity of material was too small to make the result trustworthy.

It was also attempted to estimate roughly the fusibility, by placing equally heavy beads of the pure and the commercial aluminum in front of a fine blowpipe jet, this jet being fixed in position and fed with air by a steady water blast. From the difference of distance at which the beads could be just fused it seemed probable that the pure metal was a little less fusible than that of commercial character.

Under the same conditions of heating the former seemed to oxidize on the surface with rather more ease than the latter. On the other hand, the pure metal seemed to present perceptibly greater resistance to the prolonged action of solvents—acids and alkalies—than the impure.

UNIV. OF VIRGINIA,
July 15, 1882.

CONTRIBUTIONS FROM THE LABORATORIES OF THE SCHOOL OF MINES, COLUMBIA COLLEGE.

XXXI.—ON NITRO-SACCHAROSE.

By ARTHUR H. ELLIOTT, Ph. B., F, C. S.

This curious body has already been investigated by Sobrero in 1847,* Schoenbein,† Ad & W. Knop‡ and others; and no definite conclusions as to its constitution appear to have been brought out by their labors. Having to make some experiments upon this body I thought it would not be uninteresting to give an account of some results which I obtained in the course of my work. These experiments include the action of a number of solvents, the action

* Comptes Rendus Vol. 24, p. 247.

† Pogg. Ann. Vol. 70, p. 100.

‡ Jour. fur prak. Chem, Vol. 56, p. 334.

of ammonium sulphydrate, and also the action of a strong solution of ferrous chloride.

In the preparation of nitrosaccharose, pure crystals of cane sug ar wer used and reduced to a powder fine enough to pass through a sieve with 100 holes to the linear inch, after which it was dried in a water-bath. A number of methods were used to nitrate the sugar, and a brief description of these may prove useful. In the first case the following method was used. 50 grms. of sulphuric acid (sp. gr. 1.84) and 25 grms. of nitric acid (sp. gr. 1.53) were mixed together, and the mixture cooled to 15° C.—25 grms. of the finelypowdered sugar were now slowly stirred into the acid mixture, the temperature rose to 30° C.,the mixture being constantly stirred for 15 minutes. The clot thus formed was thrown into cold water (about 1 litre) and the acid mixture was also added. The nitrosaccharose thus formed was collected together into one mass, and after washing and squeezing in plenty of cold water it weighed 24.4 grms.

The material thus obtained was a soft, silky looking, wax-like body, plastic, and adhering to the fingers when removed from water. At 30° C. it is soft enough to flow like melted pitch. It could not be washed free from acid. When heated on a water-bath it melted and began to decompose giving off red fumes. An attempt was made to neutralize it with potassic carbonate, but the action was very energetic and most of the material dissolved in water.

Another lot of nitrosaccharose was made using 25 grms. of sugar 400 grms. of sulphuric acid (sp. gr. 1.84) and 150 grms. of nitric acid (sp. gr. 1.53) following the same method of adding the sugar as mentioned before. The mixture had a temperature of 15° C. before adding the sugar and rose 10° C. during the experiment. In this case the acid mixture containing the clot of nitrosaccharose was poured into cold water and the mixture of acids and water became so hot that red fumes were evolved. The resulting mass after washing and squeezing weighed only 22 grms. The mass was melted on a water-bath under water, and while in this condition potassium carbonate was added and the whole well stirred. It was then washed from excess of alkalies and allowed to stand in water several days, changing the water daily. After a time it began to give an acid reaction, showing decomposition. It was washed with a little alcohol and allowed to dry.

A third lot of nitrosaccharose was made, using the same acids and sugar in the following proportions: sugar, 22 grms., sulphuric

acid, 176 grms., nitric acid, 66 grms. The sugar was added as usual, and the resulting mixture was poured into water in small portions, cooling the water after each addition; the nitrosaccharose thus obtained after washing and squeezing weighed 27 grms. It was washed with water and a weak solution of potassic carbonate.

The rest of the treatment was exactly like that followed in the second case.

Yet another quantity of nitrosaccharose was made, using a large quantity of sugar. Eight hundred grms. of sulphuric acid (sp. gr. 1.84), and 300 grms. of nitric acid (sp. gr. 1.53), were used with 95 grms. of sugar. When half the sugar had been added to the acid mixture, the whole was cooled, the rest of the sugar added and the completed mixture allowed to stand one hour with frequent stirring. The acids were now poured off the nitrosaccharose, and the latter, when washed and kneaded thoroughly with water, weighed 135 grms. When the mass was first removed from the acid mixture it had the consistence of soft butter, but as the washing and squeezing progressed it became more and more resinous and white. A further kneading in water hot enough to bear the hand in it, using two lots of about one litre each, and finally in water containing one per cent. of potassic carbonate, reduced the well-squeezed mass to 109 grms.

This last method appears to give the best result in the matter of washing, but the material slowly decomposes, the water in which it is kept becomes acid and smells faintly of hydrocyanic acid. Nevertheless the nitrosaccharose made by either of the methods changes so slowly that I have kept it under water for over a year without any great change in appearance or bulk, beyond the acid reaction mentioned above.

Action of Solvents on Nitrosaccharose.—The following solvents were used upon nitrosaccharose, both at ordinary temperature and also by heating in a water-bath:

Water—*Cold*, soluble; *hot*, soluble.
Absolute alcohol—*Cold* and *hot*, soluble.
Alcohol, 93 per cent.—Same as absolute.
Alcohol, 80 per cent.—*Cold*, slowly soluble; *hot*, soluble.
Alcohol, 50 per cent.—*Cold*, insoluble; *hot*, soluble.
Ether—*Cold* and *hot*, soluble.
Chloroform—*Cold* and *hot*, soluble.
Benzole—*Cold*, insoluble; *hot*, slowly soluble.
Carbon disulphide—*Cold*, insoluble; *hot*, insoluble.

Sulphuric acid (sp. gr. 1.84)—*Cold* and *hot*, soluble.
Acetic acid (glacial)—*Cold* and *hot*, soluble.
Turpentine—*Cold* and *hot*, insoluble.
Wood naphtha—*Cold* and *hot*, soluble.
Amylic alcohol—*Cold*, partly soluble; *hot*, soluble.
Carbolic acid—*Cold*, partly soluble; *hot*, soluble.
Nitrobenzole—*Cold* and *hot*, soluble.
Toluol—*Cold*, insoluble; hot, partly soluble.
Petroleum naphtha (71°–76° B'e)—*Cold* and *hot*, insoluble.
Astral oil—*Cold* and *hot*, insoluble.
Mineral sperm—*Cold* and *hot*, insoluble.
Olive oil—*Cold*, insoluble; *hot*, soluble.
Sperm oil—*Cold*, insoluble; *hot*, soluble.
Potassic hydrate (1 in 10)—*Cold*, insoluble; *hot*, partly soluble.
Ammonia hydrate—*Cold*, insoluble; *hot*, partly soluble.
Nitric acid (conc.)—*Cold* and *hot*, soluble.
Hydrochloric acid (conc.)—*Cold*, partly soluble; *hot*, slowly soluble.
Stannous chloride, decomposes it.
Ferrous sulphate, decomposes it.
Ferrous chloride, decomposes it.
Ammonia sulphydrate, decomposes it.
Borax (5 per cent. solution)—*Cold* and *hot*, insoluble.
Acetic ether—*Cold* and *hot*, soluble.
Acetone—*Cold* and *hot*, soluble.
Glycerine—*Cold*, insoluble; *hot*, soluble.
Nitroglycerine—*Cold*, slowly soluble; *hot*, readily soluble.

While trying the action of these solvents the following facts were noticed. When heated with water it decomposes as it dissolves. On heating with turpentine a slightly yellow fluid is obtained. Carbolic acid is reddened, both cold and hot. Toluol, both cold and hot, gives a red color. With olive oil mutual decomposition takes place on heating. Sperm oil acts like olive oil. Potassic hydrate gives a brown solution when heated. Ammonic hydrate, even in the cold gives a yellowish-brown color. Nitric acid, decomposes it on heating giving red fumes. Hydrochloric acid, both cold and hot decomposes nitrosaccharose. Stannous chloride is decomposed giving a white precipitate, even in the cold. Ferrous sulphate, also chloride, decomposes nitrosaccharose, even in the cold, giving brown precipitates. Ammonic sulphydrate, both hot and cold, decomposes this body, giving a liver colored solution, with separation of sulphur.

Action of Ferrous Chloride upon Nitrosaccharose.—Solution of ferrous chloride was prepared 10 c.c. of which contained 1.4 grms of iron in the ferrous state. Nitrosaccharose was dissolved in 93 per cent. alcohol and the solution mixed with ferrous chloride solution mentioned above. After heating on a water-bath excess of potassic hydrate was added, the solution filtered and the precipitate washed. Two samples of this solution were taken ; one was used to estimate glucose direct with Fehling's solution ; the other was heated with acid to invert the sugar, and then, after neutralizing, the glucose was determined as in the first case. They both gave 11.7 per cent. of glucose on the original amount of nitrosaccharose taken, showing that, as might have been expected, heating with ferrous chloride solution inverts all the sugar in nitrosaccharose. This action should be compared with the reducing action of ammonic sulphhydrate mentioned further on. It should be mentioned here that the ferrous chloride contained no free acid, since it was made by adding a large excess of iron to hydrochloric acid and heating till action ceased, a large quantity of iron remaining undissolved which was left in the solution to keep it saturated.

To determine the amount of oxidation produced by the nitrosaccharose upon the ferrous chloride, several experiments were made. In the first case the nitrosaccharose was treated with ferrous chloride and hydrochloric acid in an atmosphere of carbonic acid. The excess of ferrous chloride remaining after the action, was determined with standard solution of potassic dichromate. .705 grm. of nitrosaccharose oxidized .583 grm. of iron, which is equal to 30.6 per cent. of NO_3 in nitrosaccharose. In another experiment the nitrosaccharose was dissolved in ether and the ether solution was shaken up with pure calcic carbonate. After filtering, the calcic carbonate was washed with ether and the filtrate and washings evaporated to dryness. The residue was washed with water and contained only a trace of calcic nitrate, hence nitrosaccharose contained little free acid. The water-washed residue was now dissolved in ether and treated with ferrous chloride solution and hydrochloric acid on a water bath, the excess of ferrous salt being determined with potassic dichromate as in previous experiment. .572 grm. of nitrosaccharose oxidized .645 grm. of iron which is equal to 41.6 per cent. of NO_3 in nitrosaccharose. Another experiment in which the treatment with calcic carbonate was omitted and the ether solution treated at once with ferrous chloride and hydrochloric acid, gave the following results : .563 grm. of nitrosaccharose oxi-

dized .633 grm. of iron which is equal to 41.3 per cent. of NO_3 in nitrosaccharose.

From these experiments it appears that the body under consideration acts as a nitrate and contains but a very small quantity of free acid when thoroughly washed and squeezed. In treating with ferrous chloride direct without solution in ether, some of the nitrosaccharose is decomposed without oxidizing the iron solution.

Action of Ammonic Sulphhydrate upon Nitrosaccharose.—For these experiments the nitrosaccharose was dissolved in 93 per cent. alcohol and treated with ammonic sulphhydrate, which was made by saturating ammonic hydrate with sulphuretted hydrogen. In each case a weighed quantity of nitrosaccharose was dissolved in the alcohol, and a large excess of ammonic sulphhydrate was added to the solution. The mixture was then placed on a water bath and heated till a dry residue was left, which was treated with water, and filtered. The insoluble matter was examined and found to be sulphur. The solution was heated directly with Fehling's solution and gave only a trace of cuprous oxide, showing that the sugar had not been inverted during the reduction. This fact was established by several such experiments. Several experiments were now made by reducing the nitrosaccharose in alcoholic solution with ammonic sulphhydrate, as before stated, and then inverting the sugar by heating two hours with a little sulphuric acid. By this means the nitrosaccharose gave 28.4, 28.7, 26.5, 24.9 per cent. of cane sugar. The first and second results are probably correct, as the third and fourth results gave solutions which were colored, apparently from formation of caramel from over-heating. The acid solution obtained by pouring the mixture of sulphuric and nitric acids, after treating the sugar, in water, was kept and a special examination was made for oxalic acid. For this purpose 50 c.c. of the diluted solution were neutralized with ammonic hydrate and solution of calcic chloride added. After a time a precipitate was formed which proved to be calcic sulphate, but no calcic oxalate could be detected in it.

From a consideration of the foregoing experiments the following conclusions may be drawn: Ferrous chloride in presence of hydrochloric acid reduces nitrosaccharose and converts the sugar into glucose. Ammonic sulphhydrate reduces nitrosaccharose without inverting the sugar, or only to a very slight extent. In regard to the experimental determination of sugar and NO_3 in nitrosaccharose: if we take the percentage of sugar found and add it to the

percentage of NO_3 found by oxidizing ferrous chloride, we obtain the following results: The average of the first and second determinations of sugar by reducing with ammonic sulphhydrate (28.4 and 28.7) gives 28.55 per cent. of cane sugar in nitrosaccharose. The average of two determinations of NO_3 in nitrosaccharose (41.6 and 41.3) gives 41.45 per cent. of NO_3 in that body. Adding these together we have 70.00 per cent. as a total; the balance of the percentage consists of water, some free acids, and probably other compounds resulting from the action of the acids upon the sugar. Calculating the determined constituents to per cent., we have cane sugar, 40.78, to NO_3 59.21, or a ratio of 1 molecule of cane sugar to 8 molecules of NO_3 (exactly 1 to 8 009) From these figures it would appear that nitrosaccharose is formed by replacing eight atoms of hydrogen in a molecule of sugar by eight molecules of NO_3, giving the formula $C_{12}\ H_{14}\ (NO_3)^8\ O_{11}$. Assuming that this is the correct formula for nitrosaccharose, the theoretical percentage yield of its constituents would be sugar 41.20, NO_3 59.75. When we compare these figures with those actually obtained above, and remember that the methods used are not capable of great exactness in results, it appears most probable that the above is the true formula for nitrosaccharose.

XXXII.—NOTES ON BONE OIL.

By Arthur H. Elliott, Ph. B., F. C. S., and Ferdinand Sands, A. B., Ph. B.

During the Spring of 1881, Messrs. C. P. Sawyer and W. W. Share, two students of the class of 1881, of the School of Mines, at the suggestion of Prof. C. F. Chandler, undertook to investigate bone oil, and repeat some of the work of Anderson,* C. G. Williams,† Vohl,‡ Church, and Owen§, Schwanert,‖ Unverdorben¶ and Runge.**

**Phil. Mag.*, (3) vol. xxxiii. p. 174; (4) vol. ii. p. 457; (4) vol. ix pp. 145 and 214. *Trans. Roy. Soc Edin.*, vol. xvi. p. 4; vol. xx. (2) p. 247; vol. xxi. (1) p. 219; vol. xxi. (4) p. 571.

†*Chem Soc. Jour.*, (Lon.), vol. vii. p. 97. *Proc. Roy. Soc.*, vol. xiii. p. 305; vol. xii. p. 311.

‡*Jahresb.*, 1859, p. 742.

§*Jahresb.*, 1850, p. 259. *Phil. Mag.*, (4) vol xx. p. 110.

‖*Am. Ol. Phar...* vol. cxx. p. 279.

¶*Pgg. Ann.*, vol. xi. pp. 59, 67; vol. viii. pp. 258, 480.

***Pogg. Ann.*, vol. xxxi. pp. 65, 67. *Chem. Gaz.*, 1855, p. 308.

For this purpose they took five barrels of bone oil and submitted it to distillation, a process whose disagreeable nature none can appreciate but those who have had the task. These hard working gentlemen succeeded in obtaining a number of interesting results from the various distillates, and it is to be hoped that some day they will publish an account of them. From the five barrels of bone oil they obtained (after losing about one and a half barrels by foaming) about ten gallons of light oil and water. On treating this light oil with sulphuric acid they obtained a reddish gelatinous mass which they placed on one side, and continued their work upon other products of the bone oil.

During last winter we thought it would be interesting to find out the character of the red gelatinous body, mentioned above, obtained by treating the light oil from bone oil with sulphuric acid. We therefore took some of the distillate obtained by Messrs. Sawyer and Share and treated it with sulphuric acid. Two litres of this distillate, were mixed with about twice the volume of water, 500 c. c. of concentrated sulphuric acid were added with constant stirring, and the whole allowed to stand twenty-four hours. At the end of this time a heavy granular reddish-yellow precipitate had separated, which was strained out through muslin and squeezed in a screw press to remove acid and oil. It was thoroughly washed with water, soaking many hours, between each new addition of wash water, and it gradually assumed a darker color, finally becoming a dark brownish-black. The liquid strained from the precipitate obtained above, and containing oil, was again treated with 500 c.c. more of concentrated sulphuric acid, well agitated, and allowed to stand twenty-four hours. Another quantity of the reddish yellow precipitate was found and separated as before; it also turned brownish-black after a time. The strainings from the second precipitate were treated with sulphuric acid, and more precipitate was obtained. A fourth treatment of the strainings gave but a little of the precipitate and the treatment was discontinued. During this treatment the oil became smaller in quantity and at last there was practically nothing left from these operations but the various precipitates and the strongly acid fluid with a little oil floating on the surface.

The original light oil had a specific gravity of 0.8354 at about 20°C. and boiled at about 120°C.,at which temperature it is distilled very rapidly. This oil does not mix with water and is soluble in alcohol.

The brownish-black precipitates obtained above were all washed with ether, alcohol, and finally with hot water, and dried in a water bath. As various quantities of acid were added to the successive strainings, the precipitate became more densely gelatinous and of the consistence of leather that has been burned. But by treating with ether, alcohol, and hot water, it was found that all the precipitates could be reduced to the same black powder. This black powder was heated with the following solvents without effect: Alcohol, methyl alcohol, turpentine, glacial acetic acid, sulphuric acid, petroleum-naphtha, phenol, and glycerine, ether, chloroform, amyl alcohol, nitrobenzol, acetic ether, acetone, toluol, and carbon disulphide. If fuming nitric acid is dropped upon it violent deflagration takes place; but by pouring an excess of this acid upon the black powder, no action takes place immediately; only after long standing complete solution takes place. This solution, when diluted and excess of ammonic hydrate added, gives a bright scarlet solution, but no precipitate.

We tried the action of these solvents in the hope of being able to crystallize this body, and finally we gave up the idea. We therefore treated it again with ether, alcohol and hot water, which gave us colored solutions in each case. On evaporating these solutions we find that only trifling quantities of oily matters were left; which, in the case of the ether solution, itself of a dark orange color, gave a slight green fluorescence before evaporation.

After careful purification as above stated, an analysis of the powder was made. Much trouble was encountered in determining the carbon and hydrogen, owing to the fact that the body was found to contain considerable sulphur. But we finally succeeded in getting duplicate analyses that were satisfactory. For this purpose we mixed the black powder with a large quantity of fused lead chromate in fine powder; the gases were then made to pass over granulated copper oxide and about four inches of clean copper gauze recently reduced in hydrogen.

The nitrogen was determined by heating with soda-lime, and gave very satisfactory results.

In the case of the sulphur we had another set of failures; the fine condition of the powder causing most violent action, with various oxidizing agents. We finally succeeded in getting fair results by fusing with a mixture of twelve parts of mixed carbonates (7 of potassic carbonate to 5 of sodic carbonate), and three parts of potassic nitrate, to one part of the powder. By this means, using

a moderate heat, we succeeded in getting a quiet fusion with very gentle deflagration. Care must be taken that the fusion mixture is dry and finely powdered, and that the black powder is very intimately mixed before heating for fusion.

We made many determinations of the various constituents of this curious body, many of them approaching the results given below, which are those obtained after considerable experimenting on the various methods used for its analysis.

The following is the percentage composition of the brownish-black powder resulting from the change of the orange-red precipitate obtained by heating the bone-oil distillate with sulphuric acid:

	I.	II.
Carbon	59.56	59.44
Hydrogen	6.28	6.22
Nitrogen	11.90	11.90
Sulphur	3.92	4.15

Anderson and Schwanert both analyzed this body, but no mention is made of the presence of sulphur in it. Anderson's figures are 71.98 carbon, 6.88 hydrogen, 13.59 nitrogen; while Schwanert, who made the body from carbopyrrolic acid, found smaller quantities of carbon and nitrogen. We make these statements since we believe this body is what is generally known as "pyrrol red," and formally examined by Anderson and Schwanert with the above results. Wiedel and Ciamician* have recently worked upon this body, and have not been able to obtain it of constant composition, since they examined it in the red condition, when it changes very rapidly. The last authors do not mention the presence of sulphur in this body, and we have searched in vain for such a record. We are led to believe that pyrrol red is a compound of sulphuric acid (or hydrochloric acid when produced by that agent), with the light oil obtained from bone-oil, but cannot at present give the constitution of the body, as our work is incomplete. We would mention that by boiling with strong solution of potassic hydrate no action takes place and no perceptible quantity of ammonia is evolved; but by fusing with solid potassic hydrate after evaporation of the mixture of the black body with solution of potassic hydrate, we have obtained an oily distillate that appears very much like the original oil of the bone-oil distillate which we started with. At present we have not obtained enough of this material to examine its character.

*Berichte xiii., 65–85.

We have given these notes to call attention to our work and to see if others have not perhaps already arrived at similar conclusions. We are well aware that many others have labored hard in this field, but we have done our work with every endeavor to arrive at facts, and hope to be able to continue the investigation the coming winter, when we shall prepare this body in other ways and analyze the oil obtained by fusing with potassic hydrate.

XXXIII.—ON THE COMPOSITION OF ELEPHANT'S MILK.

By Charles A. Doremus, M. D., Ph.D.

In the spring of 1881 an opportunity presented itself for obtaining several specimens of milk from the elephant Hebe. The analysis of these samples were reported in Vol. III of the journal of the society. The exceptional character of the milk made it desirable to obtain further specimens. At the time the first investigation was undertaken two other female elephants were supposed to be with young. Such proved to be the case with one only.

The elephant Queen gave birth to a female calf at Bridgeport, Connecticut, on February 22, 1882, and the new baby was named after its birthplace. My intention was to collect some of the first milk secreted, but, though unsuccessful in this, I procured a quantity about thirty-six hours after the birth of the calf. This milk still possessed the qualities of colostrum.

The colostrum collected presented the appearance of thin milk. On standing it separated into three layers—the cream uppermost, then the usual blue layer, and at the bottom a yellow stratum composed mostly of mucous cells held together in a glairy fluid. These cells were uniform in size and in comparison with the oil globules not of that relative size usual in colustrum corpuscles. Of those peculiar bodies there were indeed few, unless these smaller cells represented them. The fat globules were not uniform in size, nor did they present that peculiar aggregation noticed in the milk from the elephant Hebe, or later that of Queen.

As the presence of albumen is one of the characteristics of colostrum, an attempt was made to determine its quantity, as well as the quantity of casein. The task proved difficult. At first Hoppe-Seyler's method was adopted, viz., that of acidulating the milk, greatly diluted, nearly to the point of coagulating the casein and then completing the reaction by saturating the liquid with CO_2. A flocculent

deposit was obtained after very long standing, but though the test was repeated several times no complete filtration could be effected. Qualitatively the experiment was successful ; the presence of albumen was readily detected in the filtrate on boiling.

As Tolmatscheff* claims that Hoppe-Seyler's method proved unsuccessful in his hands when applied to milk from women and advises the use of magnesium sulphate to saturation to precipitate casein, and alcohol to precipitate casein and albumen together, experiments were made to test its value in this instance.

By the use of magnesium sulphate the casein was separated, but the milk became so thick and mucilaginous that though left to filter during two days so viscous a mass remained on the filter that all attempts to wash it with magnesium sulphate solution proved futile. The filtrate, what there was of it, was clear and gave a coagulum when heated. The coagulum seemed more copious than that in the filtrates by Hoppe-Seyler's method, but this may have been due to the greater concentration of the liquid.

Not being able to separate the casein by these methods in such form as to weigh it, 20 cc. of the milk were treated by Hoppe-Seyler's method and the albumen determined in 100 cc. of the filtrate. The combined percentages of casein and albumen were then ascertained, which, less the percentage of albumun already found, gave that of the casein. Alcohol proved to be the best precipitant for casein and albumen. The casein separated in fine flocks, carrying albumen and fat with it. The latter was subsequently removed by ether. The liquid filtered nicely, and the precipitate could be thoroughly washed on the filter. The usual method of precipitating albumen and casein by heat and acetic acid gave a precipitate that could not be separated by filtration. Three specimens of milk were collected. Most of the determinations were duplicated. The methods of analysis was the same as that followed in the case of the milk from the elephant Hebe, except as regards albumen and casein. It was not found necessary to dry the milk in vacuo. Once, with the colostrum, sand was mixed with the milk to aid dessication.

The sugar when determined by Fehling's solution volumetrically in the colostrum and No. II. was too low, but estimated gravimetically in No. III. it agreed closely with the figures in the table. In No. I. the milk treated by alcohol and filtered left on evaporation a residue, less ash, equal to 8.0101 per cent. of sugar.

*v. Gorup-Besanez Physiologische Chemie, pp. 418 and 420.

	I. Feb. 24th. Morning.	II. April 9th. Afternoon.	III. April 22d. Afternoon.	IV. Milk from Hebe.
Quantity.....	335 cc.	420 cc.	120 cc.	72 cc.
Cream.....	7.7 vol. %	7.7	33	62
Reaction.	Alkaline.	Slightly Acid.	Neutral.	Slightly Acid.
Sp. gr. at 60° F....	1,036	1,036	1,028	1,023.7
	In 100 Parts by Weight.			
Water.....	86.5930	82.2390	76.9930	66.697
Solids.....	13.4070	17.7610	23.0070	33.303
Fat.....	3.0100	7.0576	12.6175	22.070
Solids not fat....	10.3970	10.7034	10.3895	11.233
Casein	2.0676	5.7060	5.7590	3.212
Albumen.....	0.3455			
Sugar*.....	7.6533	4.5494	4.1510	7.392
Ash.....	0.3306	0.4480	0.4795	0.629
Taste.....	Disagreeable	Much improved.	Excellent.	Excellent.
Odor...	Unpleasant	Pleasant	Pleasant	Pleasant
Color.....	Yellowish white	White and opaque	White and opaque	White and opaque
Consistency.....	Yellow deposit, otherwise fluid	Very fluid	Fluid	Fluid

Specimen No. III. was drawn after the elephant had been exhibited in the circus circus-ring and while the calf was nursing at the other teat. The calf had not nursed for some hours. Of all it approaches nearest in composition to the milk from Hebe. The solids not fat, remain constant throughout, while the fat varies considerably. Apparatus to serve as a breast pump was constructed in order to collect the milk from one teat or both during say half a day, in order to gain a knowledge of the quantity secreted daily, but the plan unfortunately could not be carried out. "Bridgeport" grew quite as rapidly as the other "baby," more than doubling her weight during the period of these experiments.

The character of the fat in the last specimen was like that obtained from Hebe, the size and aggregation of the globules and the odor of the fat after extraction being the same. During the last month's of Hebe's period of lactation the employees about the circus frequently partook of her milk and found it most palatable.

*By difference.

REVIEW OF PAPERS ON ANALYTICAL CHEMISTRY (INORGANIC) WHICH HAVE APPEARED DURING THE YEAR 1882.

By E. Waller, Ph. D.

A modification of the apparatus for the absolute DETERMINATION OF NITROGEN, invented by S. W. Johnson and E. H. Jenkins, is described by *T. S. Gladding* (*Am. Chem. Jour.*, IV., 42).

According to *A. Guyard* (*Chem. News*, XLV. 159), ALL ACIDS AND OXIDES OF NITROGEN are converted into ammonia by combustion with marsh gas and soda lime. The mixture is made in the proportion of 75 grs. of sodium acetate with 1½ oz. of soda lime, and the combustion conducted in the ordinary manner. The tube is charged first with about one-quarter of the above mixture, then the remainder mixed with 5 to 7 grms. of the substance to be examined is added, and finally pure soda lime.

The accuracy of the results thus obtained is disputed by *J. Ruffle*, (*Chem. News*, XLV., 186).

In experimenting on methods for the DETERMINATION OF NITROUS OXIDE, *W. Hempel* (*Ber. D. Chem. Ges.*, XV., 903 *et seq.*) finds it extremely difficult to obtain absolutely pure N_2O. The purest gas obtained contained 98.8%. He finds the best method of estimating the gas to be by explosion with hydrogen in an apparatus specially designed for the purpose, which is carefully described.

100 vols. N_2O with 460 vols. H was not explosive, while 100 N_2O with 116 vols. H caused such a violent explosion as to shatter the apparatus. The mixture therefore must be made within these limits.

Alkaline solutions of potassium permanganates or of chromic acid are recommended by *C. Boehmer* (*Fres. Zeits. Anal. Chem.*, XXI., 212,) as ABSORBENTS FOR N_2O_2 in analytical processes.

To determine the amounts of sodium SULPHIDE, SULPHITE AND THIOSULPHATE in the same solution, *G. E. Davis* (*Jour. Soc. Chem. Ind.*, I., 89), determines, 1. The amount of iodine absorbed by a measured quantity of the solution, by converting the alkalies present into bicarbonates with carbonic acid water and titrating with iodine solution. 2. The amount of sulphide and thiosulphate present; by adding $SrCl_2$ in excess, digesting for about half an hour at 65° to 70° C., filtering off the strontium sulphite, washing with air free from water, adding carbonic acid water to the filtrate, and titrating with iodine solution. 3. The amount of thiosulphate

present; by separating the sulphite as in 2, and then separating the sulphide by digestion for half an hour with $CdCO_3$, filtering and titrating the filtrate with the same precautions as before. A correction must be applied to the results before calculating from the data obtained. It has been found that 100 cc. of a solution of $SrSO_3$ saturated at 65 to 70° C., consumes 0.3 cc. decinormal iodine, and the same bulk saturated at 12.7° C. consumes 0.16 cc. decinormal iodine solution. The bulk and temperature of the filtrates must therefore be taken.

To obtain the SULPHUR IN PYRITES in a form for estimation as sulphate, *Fr. Bockmann* (*Fres. Zeits. Anal. Chem*, XXI. 90), fuses 0.5 gms. with 25 gms. of a mixture containing 6 parts Na_2CO_3 to 1 part $KClO_3$.

In estimating CHLORINE GRAVIMETRICALLY, *D. Lindo* (*Chem. News*, XIV., 193) filters the Ag Cl through a Gooch filter and dries for half an hour at 140 to 150° C.

In the indirect estimation of CHLORINE AND BROMINE, *L. P. Kinnicutt* (*Am. Chem. Jour.*, IV., 22), after weighing the mixed silver salts in a platinum crucible, reduces to metallic silver by a battery, the crucible being made the positive electrode of the circuit.

The sanitary EXAMINATION OF GASES ESCAPING FROM CHEMICAL WORKS forms the subject of a paper by *W. J. Lovett* (*Jour. Soc. Chem. Ind.*, I., 210). For *nitrous compounds* the gases are brought into contact (by agitation) with hydrogen peroxide and water, the solution neutralized with pure soda solution (free from nitrates) evaporated to very small bulk, and after removing carbonic acid by a drop of sulphuric acid, using a Davis nitrometer tube with conc. H_2SO_4 and mercury, the volume of N_2O_2 being measured. For *sulphurous acid* the gases may be passed through a standard solution of potassium permanganate. For *oxygen*, the gas is caught in a U tube, one limb of which is sealed and graduated so as to form an eudiometer, and oxygen is absorbed by potassic hydrate and pyrogallic acid. For *sulphuretted hydrogen* the gas may be passed through $Pb(NO_3)_2$, and the precipitate converted into sulphate and weighed, or the excess of lead remaining in the solution titrated with potassium dichromate. For *chlorine* the gases are passed through KI solution, and the iodine set free titrated with sodium thiosulphate. To distinguish between the amounts of *free and combined chlorine* in such escapes, *G. E. Davis* (loc. cit. p. 214) passes the gases through H_2S water. The solution thus obtained is halved. One half is neutralized with $MgCO_3$, and then titrated with $AgNO_3$,

giving total chlorine ; the other half evaporated to neutrality without any addition and titrated, giving combined chlorine.

To determine the amount of OZONE IN THE AIR, *D. Tommasi* (*Les Mondes*, I. [3] 123), draws a measured quantity through a tube having two branches. The air passing through one branch is drawn directly through a standard solution of sodium arsenite ; that passing through the other branch is first passed over red-hot platinum sponge before passing into a solution of sodium arsenite. The solutions are then titrated with potassium permanganate, and the difference of the results in the two solutions serves as a basis for the calculation of the ozone present, as in the second solution the ozone has been destroyed by the ignited platinum sponge.

Experiments on potassium permanganate in relation to its application for the EXAMINATION OF POTABLE WATERS, have been made by *A. Dupre* (*Analyst* VII. 1). Water acidified with sulphuric acid when absolutely pure, does not decompose permanganate in a closed flask at 80° F. In standardizing the permanganate for Tidy's test the operation should be performed in a closed flask. With waters for domestic use which are comparatively free from organic matter, the degree of temperature and the length of time for the action of the permanganate as well as the qantity of permanganate, make but little difference in the results obtained. With less pure waters the differences between the 3 hour and 4 hour test were very decided. On using phosphoric acid instead of sulphuric in Tidy's test, results much lower were obtained. The addition of sulphuric acid and boiling before adding permanganate, increases the amounts of oxygen absorbed. This increase was very marked when the test was tried on pure water, to which cane sugar, starch, or urine had been added, but was much less marked when gelatine or egg albumen was used. *C. W. Marsh* (*Am. Chem. Jour.* IV. 188) finds that in many waters examined, the distillate obtained by distilling with sodium carbonate, affords more ammonia (determined by the Nessler) on redistilling with alkaline potassium permanganate than if the Nessler test is directly applied to it.

The ORGANIC NITROGEN in waters, beer, etc., is estimated by *W. Bettel* (*Chem. News*, XLV. 38), in one operation by distilling from a copper flask in a current of hydrogen with pure soda (from sodium). The use of the copper flask admits of distillation to complete dryness, and ignition of the residue. At the close of the operation to break up cyanides, &c., which may have formed, a little potassium permanganate is added, and heat again applied. Nitrates, if

present, are previously converted into ammonia by the action of the zinc-copper couple. The distillate contains all the nitrogen in the form of ammonia.

To convert NITRATES AND NITRITES IN WATER into ammonia, *F. P. Perkins* (*Analyst* VII. 66,) proposes the use of metallic magnesium aided perhaps by the addition of some Na Cl. *J. B. Kinnear* (*Chem. News*, XLVI. 33) finds that zinc with a dilute acid will effect the reduction in about ten minutes, provided that there are about 5,000 parts of liquid present for every part of nitric nitrogen. The rapidity of the reduction depends somewhat upon the amount of surface of zinc exposed.

Among delicate TESTS FOR NITRITES in water, *A. Jorissen* (*Fres. Zeits Anal. Chem.* XXI. 210) proposes a solution containing 0.01 gm. fuchsin in 100cc. of glacial acetic acid, which gives a change of color, passing through blue and green to reddish yellow. (Noted by Vogel *Jour. Prakt. Chem.* XCIV. 457), while *E. W. Davy* (*Chem News* XLVI. 1) recommends for the same purpose acidifying the solution, adding a few cc. of gallic acid and boiling. The presence of nitrites at once gives a tint to the water similar to that of the Nessler test with ammonia. The reaction is stated to be :

$$C_7 H_6 O_5 + 2 H N O_2 = C_6 H_4 O_3 + C O_2 + 2 N O + 2 H_2 O$$

tanno-melanic acid being formed.

The COLOR METER FOR WATERS used by Messrs. *Crookes, Odling, and Tidy* in their examination of the London water (*Report to Local Government Board for* 1881) is described (*Chem. News* XLV. 170). The two-foot tube is used. For comparison colors, two wedges, one brown, the other blue, are used, which are slid over one another until a tint corresponding to that in the water is obtained. The solutions contain respectively :

Brown, 0.7 gm. metallic iron as ferric chloride
0.3 " " cobalt as chloride,

and a slight excess of free acid per litre.

Blue, 5 gms. pure crystallized cupric acid sulphate per litre. The results are recorded in figures representing the thickness in millimetres of the solutions required to simulate the color of the water thus : Brown : Blue, 20 : 10.

Regarding the examination of fertilizers for REVERTED OR RETROGRADE PHOSPHATES, *C. Richardson* (*Am. Chem. Jour.* IV. 183) finds that the use of ammonium oxalate for their extraction gives variable results depending upon the time and temperature of diges-

tion, the degree of concentration of the solution, &c. In this connection see also results of *T. S. Gladding* (this *Journal*, p 113). *F. J. Lloyd* (*Jour. Lond. Chem. Soc.* XLI. 306) triturates 0.5 gm. of the fertilizer with 20cc. of a solution containing 30 per cent. citric acid, and enough ammonia to render it strongly alkaline. The mixture is made up to 100cc. and allowed to stand over night at the temperature of the laboratory. In 50cc. of the clear solution the lime is separated by acidifying with acetic acid and adding ammonium oxalate, and in the filtrate the phosphoric acid is determined by the use of magnesia mixture.

As to VOLUMETRIC processes for PHOSPHORIC ACID, *C. Mohr* (*Fres. Zeits. Anal. Chem.* XXI. 216) shows by a series of experiments that even small amounts of ammonia salts have a marked effect on the results obtained by the uranium titration, while *H. Pemberton* (*Jour. Franklin Inst.* CXIII. 184). proposes the use of a solution of neutral ammonium molybdate containing 89.543 gms. of the salt per litre (1cc=0.003 gm. $P_2 O_5$). For titration 100 to 125cc. of the solution is taken [which should not contain over 0.1 gm. $P_2 O_5$] 10 gms of ammonium nitrate, and 2cc. of nitric acid [sp. Gr. 1.14] are added and the solution is heated to 140° F. or over. The end reaction is the absence of further precipitation. Toward the close of the titration portions of the solution have to be filtered to give opportunity to observe the end reaction. If the titration is overrun, a measured quantity of $Na_2 H PO_4$ solution of known strength is added and the titration resumed. From the burette reading 0.5cc. is to be deducted for the effect of the nitric acid added. Silica and organic matter cause erroneous results when present, while chlorides, iron [to the extent of 0.1 to 0.3 gm.] &c., do not.

In the EXAMINATION OF TITANIFEROUS IRON ORES, *T. M. Drown and P. W. Shimer* (*Am. Chem. Jour.* IV. 1), treat the ore (2 to 5 grms.) with 50cc. of H Cl. (Sp. Gr. 1.12) evaporate to dryness and heat in the air bath for an hour, add 50cc more H Cl., heat and filter; turbidity in the washings is prevented by the use of ammonium nitrate. The phosphoric acid is extracted from the residue by fusion with sodium carbonate, extracting with water, evaporating to dryness with nitric acid to remove silica, &c. Molybdate mixture may then be added to the solution. In the first solution (obtained by H Cl.) the H Cl. is removed by evaporation with nitric acid, when after partially neutralizing, molybdate solution is added. This last molybdate precipitate requires purifying by dissolving in ammonia, treating the undissolved residue with nitric acid and pre-

cipitating that solution with more molybdate. After obtaining all the ammoniacal solutions of the phospho molybdate free from silica, titanic acid, &c., they are united and precipitated as usual with magnesia mixture.

For TITANIC ACID these gentlemen decompose the ore by fusion with $K HSO_4$ and after neutralizing and reducing with SO_2 precipitate by boiling as usual. *P. T. Austen* and *F. A. Wilber* (*Am. Chem. Jour.* IV. 211) find that the addition of 50 to 60cc. of acetic acid aid materially in the precipitation of the titanic acid, giving good results on boiling, even when chlorides are present. The last named decompose the ore by fusion with alkaline carbonates and treating with HCl. evaporating, &c.

For the *determination* of CARBON AND SILICON IN IRON, STEEL, etc., *F. Watts* (*Chem. News*, XLV., 279) gives the preference to a modification of Wohler's method.—Volatilization of the iron in a stream of Cl gas and making a combustion of the carbon remaining. Two operations are required for each analysis. In the first after volatilizing the iron in a current of *dry* chlorine free from oxygen, the residue is submitted to combustion, and the *total carbon* is thus determined. In the second the residue (after volatilizing iron) is weighed, giving *total carbon and slag*, while the gases from this operation are passed into water, where the $Si Cl_4$ is decomposed, and by boiling, evaporating to dryness, etc., the $Si O_2$ corresponding to *combined* silicon is obtained.

SODA COMPOUNDS may be estimated in potassium carbonate, according to *A. Van Hasselt* (*Jour. Soc. Chem. Ind.* I, 203), by treating a weighed quantity (converted into chloride) with H Cl. (Sp. Gr. 1.189) saturated with Na Cl.

To estimate CAUSTIC ALKALI IN PRESENCE OF CARBONATE, *R. B. Warder*, (*Sci. Proc. Ohio Mech. Inst.* I, 45) uses a process based upon the fact that phenolphthalein is colorless in the presence of alkaline bicarbonates. On titrating a very dilute solution of the sample (cold) until the phenol pthalein which has been added imparts no color to the solution, all the caustic and half of the carbonated alkali is neutralized, giving a result A. Then by boiling and titrating the remainder of the alkali is neutralized; result B. A+B measures total alkalinity, and A—B measures caustic alkali

To detect and separate ALKALIES IN ARGENTIC NITRATE. *M Stolba* (*Jour. de Pharm. et de Chim.*) uses hydrofluosilicic acid. By the use of phenol phthalein *M. Richter* (*Fres Zeits. Anal. Chem.*, XXI, 205) determines acidimetrically the CHROMIC ACID IN

POTASSIC DICHROMATE. Many other indicators cannot be used on account of the oxidizing action of the chromic acid upon them.

By fusing hydrorufigallic acid with potassa A NEW INDICATOR for alkalimetric titration is made by *J. Oser and W. Kalmann* (*Monats hefte f Chemie*, II, 50). It dissolves in alkalies with an olivegreen tint—a slight excess of alkali gives carmine red, changed to yellow by free mineral acids. It is unaffected by CO_2.

To DETERMINE ZINC in its ores, *A. Millot* (*Bull Soc. Chim.*, XXXVII, 339) recommends dissolving with HCl and $KClO_3$ evaporating, adding ammonia and ammonium carbonate, diluting the filtrate therefrom, and in an aliquot portion of this filtrate, adding a little K Cy and precipitating by the battery. The addition of ammonium acetate prevents the corrosion of the electrodes.

In TITRATING ZINC by Fahlberg's method manganese must be first separated to obtain correct results. *R. W. Mahon* (*Am. Chem. Jour*, IV, 53). In titrating with Na_2S, the use of paper soaked in thallium solution is recommended by *M. Schröder* (*Berg. u Huttenmann, Zeit.* 1882, p. 4) as an indicator. If a slight excess of ammonia is present the thallium salt is unaffected by Zn S.

To separate NICKEL FROM COBALT *G. Delvaux* (*Comptes Rendus*) dissolves in aqua regia with excess of HCl—adds ammonia in excess, then potassium permanganate until the rose color holds for some time. Solution of pure KHO will then precipitate nickel (with manganese if present), leaving cobalt in solution. In case small amounts of nickel and large amounts of cobalt are present *A. Jorissen* (*Fres. Zeits. Anal. Chem.*, XXI, 208) precipitates with NaHO, adds bromine, then a few cc. of K Cy, and agitates in the cold. The cobalt is dissolved while the nickel remains in the precipitate.

A. Carnot (*Bull. Soc. Chim.*, XXXVII, 482) ESTIMATES CHROMIUM (as base) by adding sodium acetate to the solution slightly acidified, adding an alkaline phosphate and boiling. The presence of oxalates must be avoided. The precipitate dried at 100° contains $6\ H_2O$. Ignited it consists of $Cr_2(PO_4)_2$, containing 51.86 per cent. Cr_2O_3.

In the examination of iron ores, etc., containing chromium error may be caused, according to *B. Reinitzer* (*Monats hefte f. Chem.*, 1882, p. 249), by the chromium preventing the precipitation of some of the iron and alumina on boiling the solution after neutralizing and adding sodium acetate.

For the detection of IMPURITIES IN COPPER several chemists communicate their methods—

For ARSENIC *J. Pattinson* (*Newcastle-on-Tyne Chem. Soc.*, Feb. 1882) dissolves in nitric acid; then by adding sufficient soda solution to a little more than neutralize the free acid present, the arsenic is precipitated as copper arsenate; after standing for half an hour with frequent stirring the precipitate is filtered and washed cold. The arsenic is thus concentrated in the precipitate. *A. H. Sexton* (*Chem. News*, XLV, 255) prefers to precipitate the arsenic from the nitric acid solution (by adding ferric nitrate, neutralizing, adding sodium acetate and boiling) as basic acetate. The precipitate is dissolved in acid, precipitated by H_2S, oxidized to arsenic acid, and determined as the magnesia compound.

For CADMIUM *A. Orlowski* (*Fres. Zeits. Anal. Chem.*, XXI, 214) first removes Bi if present, acidifies with HCl, decolorizes by $SnCl_2$, adds milk of sulphur and heats to boiling. This precipitates the copper as Cu_2S. From the filtrate the tin is removed by ammonia, leaving the cadmium to be detected in the solution. Another method by the same author is proposed, in which sodium thiosulphate is substituted for $Sn\,Cl_2$ and milk of sulphur. In other respects it is the same.

The same author (loc. cit.) proposes to detect CUPROUS COMPOUNDS IN PRESENCE OF CUPRIC by acidifying with HCl and boiling with milk of sulphur. As above, cuprous sulphide will precipitate, while the cupric compounds are unaffected. An elaborate scheme for the EXAMINATION OF COMMERCIAL COPPER for gold, silver, lead, bismuth, antimony, tin, arsenic, iron, cobalt, nickel, zinc, sulphur and phosphorous is given by *R. Fresenius* (*Fres. Zeits. Anal. Chem.*, XXI, 229).

M. M. P. Muir (*Jour. Lond. Chem. Soc.*, XLI, 1) determines BISMUTH VOLUMETRICALLY by separating it from other metals, as oxychloride, converting this compound into chloride, dissolving in glacial acetic acid and precipitating by excess of standardized solution of $K_2C_2O_4$. The precipitate is $K_2C_2O_4$, $Bi_2\,(C_2O_4)_3$ The excess of oxalate is titarted with permanganate.

For the VOLUMETRIC DETERMINATION OF ANTIMONY *E. T. Herroun* (*Chem. News*, XLV, 101) converts the antimony into $Sb\,Cl_5$ by treatment with HCl and $KClO_3$, boiling gently to expel excess of chlorine, cooling and adding an excess of KI. The iodine freed by the action of the $Sb\,Cl_5$ is then titrated with a standardized solution of sodium hyposulphite.

M. M. P. Muir (*Chem. News*, XLV, 69) detects TIN IN THE PRESENCE OF ANTIMONY by boiling the HCl solution for 10 minutes with copper turnings which reduces it to stannous form and permits of its being detected by $HgCl_2$.

To detect SMALL AMOUNTS OF SILVER IN LEAD (reduced from galena, etc.) *J. Krutwig* (*Ber. D. Chem. Ges.* XV, 307) dissolves in nitric acid and precipitates with large excess of NaHO. Silver plumbate remains insoluble. By extracting with ammonia, evaporating to dryness, dissolving in nitric acid and separating the lead by H_2SO_4, a solution is obtained in which the silver may be detected with HCl.

To SEPARATE GALLIUM from zinc and ferrous oxide *L. de Boisbaudran* (*Comptes Rendus*, 1882, *No.* 17) uses cupric hydrate instead of barium or calcium carbonate. The copper may afterward be separated by dissolving and adding H_2S.

ABSTRACTS.

Abstracts from *Journal of the Chemical Society*, London, by Arthur H. Elliott, Ph. B., F. C. S.

On some Higher Oxides of Manganese and their Hydrates. (Part II., Vol. XL., p. 56). BY V. H. VELEY, B. A., F. I. C.

The higher oxides of manganese, when heated in dry nitrogen at temperatures ranging from 60°—200°C., are simply dehydrated without loss of available oxygen; the dehydrated oxide formed readily absorbs oxygen. The oxides, when heated in dry hydrogen, are simultaneously dehydrated and reduced; a hydrate of the red oxide is formed which readily absorbs oxygen. The quantity of water retained by the peroxides when heated to 200°C. in dry hydrogen, is greater than that retained by the same oxide at the same heat in air, oxygen, or nitrogen.

On a New Alkaloid from Cinchona Bark. (Vol. XL., p. 66) BY DAVID HOWARD AND JOHN HODGKIN.

The alkaloid is obtained from *China Cuprea* of Dr. Fluckiger. Its specific rotary power is very near that of quinine: 100 cc. of ether free from alcohol dissolves 0.57 of it at 12°C. Ether containing 8 per cent. of alcohol dissolves 2.27. Alcohol of 90 per cent. at 12°C. dissolves 7.64 per 100 cc. When impure or mixed with

other alkaloids it is much more soluble. Authors describe the sulphate, bisulphate, oxalate, tartrate, hydrochloride, and the iodosulphate ; also the platinum salt and the action of chlorine and ammonia.

Contributions to the Chemistry of Rare Earth-Metals. (Vol. XL., p. 68). By B. Brauner, Ph. D.

The object of this research is the study of the cerite metals, with special reference to their position in the periodic system. The metals cerium, lanthanum, and didymium, are each found to occupy its own characteristic position in the system.

Note on Certain Photographs of the Ultra-violet Spectra of Elementary Bodies. By W. N. Hartley, F. R. S. E. Vol. XL., p. 84. An account of the application of the new dry plate process of photography to the production of spectra-photographs By means of prisms and lenses of quartz or Iceland spar, photographs were obtained which contained the whole of the lines of metallic spectra completely in focus on one plate, and extending from the blue to the extreme ultra-violet rays. By prolonged exposure of gelatin plates photographs to the red end of the spectrum may be obtained. The paper is illustrated with Woodburytypes of the spectra.

The Chemistry of Bast Fibres. By C. F. Cross and E. J. Bevan. Vol. XL, p. 90. Authors give an account of their researches upon the action of halogens upon jute and their fibres; more especially the action of bromine. Also the action of dilute sulphuric acid upon fibres. They are led to conclude that jute fibre is composed for the most part *not* of cellulose, but of a transition form of cellulose from that of the original carbohydrate to its ultimate modification of a soluble astringent. They call the transition modification of cellulose *Bastose*, and give the name *Bastin* to the aromatic derivatives of cellulose. Incidentally they discuss Glycolignose and Glycodrupose; the origin of tannins; reactions of the jute substance under high pressure; reduction of indigo by jute; and reaction of linseed oil with sulphuric acid. In conclusion they give a diagram showing the passage of cellulose into aromatic bodies allied to the tannins.

A New Apparatus for the Determination of Melting Points. By C. F. Cross and E. J. Bevan. Vol. XL., p. 111. A small cup is attached to the bulb of a thermometer, and in this cup

some of the body to be tested is melted. While the body is still liquid a minute bulb tube, with a piece of platinum wire fixed in at the bottom, is dipped into the liquid and allowed to stay there till the whole is solidified. To determine a melting point the combination is placed in mercury, the latter is heated, and when the body melts the bulb tube rises to the surface and the thermometer registers the melting point at that instant. Authors state that no stirring is necessary during the heating of the mercury.

On the Reaction of Chromic Anhydride with Sulphuric Acid. By C. F. Cross and A. Higgin. Vol. XL., p. 113. Authors examined the insoluble bodies formed by the action of sulphuric acid upon chromic anhydride. They find they are only approximately constant in composition, and give as an average formula $4\ Cr_2O_3 .\ 7\ H_2O .\ 13\ SO_3$, or $2\ (2\ Cr_2O_3 .\ 3\ SO_3)\ 7\ H_2SO_4$. These bodies are entirely insoluble in water, and are attacked with difficulty by solutions of alkaline hydrates. Potassium dichromate and sulphuric acid give coarsely granular precipitates that contain potassium.

On Some Decompositions Produced by the Action of Chloride of Aluminium. By C. Friedel and J. M. Crafts. Vol. XL., p. 115. Paper contains remarks upon E. H. Rennie's note (p. 33) on the action of ethyl chlorocarbonate on benzene. Authors state that they had attained exactly the same results, and described them in Compt. rend., June 18th, 1877. They are now engaged in the study of the destructive action of aluminium chloride upon hydrocarbons, and state that the higher homologues of benzene lose their side groups and tend to be resolved into benzene when heated with aluminium chloride. Hexmethyl-benzene is easily reduced to durene and simpler hydrocarbons.

Chemical Examination of the Buxton Thermal Water By J. C. Thresh, B. Sc., Vol. XL., p. 117. The density of the water at 25°.8 C. was 0.99686; and when compared with pure water at the same temperature was 0.99992. The author gives minute details of his methods of analysis in which he follows the course of procedure recommended by Bunsen. The paper contains elaborate tables showing the analyses of the portions of the total residue both soluble and insoluble in water.

For comparison with the results of other analysts the author gives his results in the following form; in which the constituents are arranged as parts in 10,000.

Temperature of water.....81.7° F
Specific gravity.....0.99992
Total solids.....2.934

Ca 0.567—Ba 0.004—Sr trace Mg 0.195—Na 0.213—K 0.040—Li trace—NH_4 0.0001—Al trace—Fe 0.002—Mn 0.0014—Pb trace—CO_3 1.238—Fl 0.0014—Cl 0.370—I nil—SO_4 0.160—NO_3 0.003—Organic matter 0.003—Si O_2 0.136—PO_4 trace.

And the following elements were detected in the sinter Mo, Cu, Co and Zn.

The gaseous constituents in cubic centimetres per litre at O° C and 760 m. m. pressure were CO_2 14.56—N 22.13.

The ammonia was determined by distilling with sodium carbonate and estimating with Nessler's solution in the distillate. The nitric acid was obtained by digesting with zinc-iron couples and then distilling and estimating the resulting ammonia by Nessler's solution. Nitrous acid was sought but not found.

Dibenzoylaniline and its Isomerides. By A. Higgin, Vol. XL., p. 132. The author has repeated the work of Gerhardt in obtaining dibenzoylaniline by the action of benzoyl chloride upon monobenzylaniline; also the work of Losanitch in obtaining phenyldibenzoylamide by the action of benzoic acid upon phenyl thiocarbimide at 130–150° C. The author states that it is necessary not to use the thiocarbimide in excess. Phenylbenzoyl-benzoylamide was prepared by acting upon phenylbenzoylamide with benzoyl chloride in presence of zinc chloride. It crystalizes in colorless plates melting at 150°; and when decomposed with dilute hydrochloric acid yields benzoic acid and a solid base, benzoylphenylamine. The sulphate of this base was made, also the chloride and a platinochloride. In the alcoholic solution from which the phenylbenzoyl-benzoylamide had crystallized an isomer of this body was obtained.

Contributions to Our Knowledge of the Composition of Alloys and Metal Work, for the Most Part Ancient. By Walter Flight, D. Sc., F. G. S. Vol. XL., p. 134.

Author gives a number of facts relating to the history of copper-nickel coinage. Coins of Ancient India 500 B. C. contained Ag 89.120—Ag Cl 1.328—Au 1.253—Cu 4.337—Pb 3.735—Graphite 0.075. A figure of Buddha contained Ag 57.701—Ag Cl 4.113—Cu 37.548—Au 0.483—Graphite 0.403. Bidrai Ware contained Zn 94.552 to 93,516—Cu 3.920 to 3.278—Pb 1.400 to 2.171

—Au nil to 0.690—Fe 0.128 to 0.345. A sickle found by Belzoni under the feet of a sphinx at Karnak, Egypt, contained: Silica 11.884—Magnetite 5.111—Calcium carbonate 0.172—Ferric oxide 64.604—Water 18.229—Nickel trace. A double hook of "Bronze" found in one of "Dixon's Passages" of the Great Pyramid contained Cu 99.521—Fe 0.479. "Bronze" figures from Egypt contained Cu 68.421—Fe 4.694—Pb 22.759—Ni (Co) 0.782—Sn 0.938—As 1.479—Sb 0.668. Another "Bronze" figure from Egypt contained Cu 82.19—Pb 15.79—Sn 2.02. Three copper spear heads from Cyprus contained :

	I.	II.	III.
Cu	97.226	98.398	99.470
Fe	1.322	0.729	0.384
Ni	—	0.153	0.084
Au	0.279	0.305	—
Pb	0.076	—	—
Sn	trace	—	—
As	1.348	trace	trace
S	—	0.305	—

All contain traces of P.

Copper axe-heads from Beth Saour, near Bethlelem, Holy Land, contained 99.557 and 99.93 per cent. of copper, with traces of As, Fe, Ni and S. Hebrew shekel contained Ag 97.674—Au 0.676—Cu 0.642—Graphite 0.034. The following bronzes : I, sword or dagger blade from Cyprus ; II, Part of figure of Mercury, of Romano-British times, from Leadenhall street, London ; III, Handle of vase, found at Island of Tilos, of about the time of Alexander the Great ; IV, Roman bronze figure in the Blacas Collection of British Museum, had the subjoined composition :

	I.	II.	III.	IV.
Cu	88.771	78.355	81.764	87.158
Sn	8.508	10.018	10.901	10.724
Pb	1.504	9.302	5.246	2.005
Fe	0.476	0.715	0.153	trace
Co	0.304	—	1.222	—

Ni, P, Au and Ag were only found in traces.

An Incas Pin, found on a mummy at Arica, S. Peru, after earthquake 1868, contained Ag 82.222—Ag Cl 1.388—Au 0.406—Cu 16.100—Fe 0.119. Bronze bar, from ruins of Temple in Bolivia contained Cu 93.26—Sn 6.52—Fe 0.26—Ni and As traces. These

results are given with a great many interesting details as to the origin and antiquity of the various objects examined.

Abstracts from *Berichte der Deutschen Chemischen Gesellschaft*, by L. H. Friedburg.

Relations between the amounts of rotation on the plane of polarization, F. Flawitzky (Vol. XV., p. 5).

Critique of Krecke's first law: "An optically active substance, forming a combination with an optically non-active one, or modified by chemical agents, either maintains its molecular power of rotation unchanged, or the latter is a simple multiple of the former." The author finds that under certain conditions and regarding substances genetically related, the relations between the molecular power of rotation are independent of concentration. Such an independence might indicate eventually the immutability of optical activity in chemical transformations.

The Molecular Power of Refraction of the Terpenes. F. Flawitzky. (Vol. XV., p. 15).—The difference of molecular refraction of the Terpenes correspond to their atomicity.

On the Action of the two Isomeric Mono-Brom-Cinnamic Acids with Conc. Sulphuric Acid. R. Leuckart. (Vol. XV., p. 16).—A remarkable difference is manifested in treating either Alpha or Beta mono brom-cinnamic acid with conc. sulphuric acid, which acts as an agent of condensation. Pure Alpha acid is hardly attacked, if at all, whilst the isomeric Beta acid is decomposed, though the products of decomposition and the constitution of these products are not yet either fully studied or known.

A constitutional similarity between these acids and fumaric and maleic acid seems possible.

Azodyestuffs. O. Wallach. (Vol. XV., p. 22).—Diazo compounds and phenols have of late been combined to a large series of dyestuffs. The general formula of these might be expressed thus:

$$R - . - N = : = N - . - Ph.$$

R meaning any monovalent radical of an aromatic compound (thus: $C_6 H_5 - . -$; $C_6 H_4 S O_3 H - . -$; $C_{10} H_7 - . -$ &c.), and Ph understood as any monatomic rest of a phenol (e. g.: $C_6 H_4 O H - . -$; $C_6 H_3 (O H)_2 - . -$; $C_{10} H_5 (S O_3 H) (O H) - . -$ &c.

Combinations with more than one azo group have been prepared only sporadically, and such with *different* azo-groups in *one* rest of phenol only once by Griess.

Wallach prepares combinations of diatomic phenols with two similar or dissimilar diazo-rests.

General formula:

$$\left.\begin{matrix} R^1 N = : = N \\ R\ N = : = N \end{matrix}\right\rangle C^6 H_3 (O H)_2.$$

In which R and R^1 may be identical or different.

Further on the question is put, whether it makes any difference, or not, in what consecutive order *different* azo-rests are incorporated into a diatomic phenol.

In pursuing those questions, the author obtains a series of new dyestuffs, the preparation of which has been patented.

On Caffein. EMIL FISCHER. (Vol. XV., p. 29).—The author continues experiments to prove the constitutional formula of his synthetic caffein, and consecutively he gives such fourmula for caffolin, hydroxy-caffein, apocaffein, hypocaffein. Finally his endeavors have resulted in producing a caffuric and hydrocaffuric acids, and theobromin.

The Action of Chloride of Aluminium on Mono-Brombenzol. O. V. DUMREICHER. (Vol. XV., p. 33).

On Gum Arabic.—HEINRICH KILIANI, (Vol. XV. p. 34). A comparison of ten different samples of gum arabic, (including two samples of arabic acid) in regard to rotatory power and percentage of mucic acid.

Occurrence of large Crystals of Ammonium Magnesium Phosphate in Urine Nearly 100 Years Old. H. SCHWANERT. (Vol. XV. p. 37). A bottle with urine was found some time ago in a coffin along with a perfectly preserved human skeleton. The coffin was found underneath a street at Wadersloh, in Westphalia by Dr. Gruwe. The street bordered an old cemetery, not used since 1830 as burial ground. In 1827 or 1828 this street was built on part of the cemetery. The bottle with urine, a green, short-necked cylindrical bottle, stoppered with a dark-brown colored cork, pressed somewhat into the neck, had most likely been put into the coffin from superstitious motives. Such superstition exists even to day in that part of the country.

The sediment of the urine consisted of microscopical white and

brown particles, the former being carbonate of calcium the latter urate of ammonium showing the murexide reaction. The three larger crystals, one of which was 8 mm. long were ammonium magnesium phosphate.

Expulsion of the Sulpho-Group by Bromine. WERNER KELBE. (Vol. XV. p. 39). Sulphurous acid, bromine and water, yield hydrobromic and sulphuric-acids.

An aromatic sulpho-acid, (which can be regarded as a substituted sulphurous acid) bromine and water, yield at a certain temperature hydrobromic and sulphuric acids, and a monobrominated hydrocarbon.

Kelbe thus prepared: Alpha—Brommeta-iso-cymol and Beta—Brommeta-iso-cymol.

Reaction of Phosgen on Diazo-Amido Compounds. A. SARAUW, (Vol. XV. p. 42). This research shows, that neighboring positions must be ascribed to: the imido-group and the tolyl-rest in diazo-benzol-para-toluidide; the imido group and the carboxyl benzol-rest in diazo—amido—benzoic acid; the imido-group and the bromphenyl-rest in diazo-benzol-parabrom-anilid. Therefore it is likely, that in the mixed diazo-amido-compounds, the imido group is combined with the more negative aromatic rest.

On Tetrabrombenzol. RICHARD MEYER, (Vol. XV. p. 46)., Bromine and nitrobenzol brought together at a high temperatures from the *same* tetra-brombenzol as is generated by the reaction between para-di-brombenzol and an excess of bromine at + 150°C.

Hence Kekule was right, in supposing that his combination, in spite of great differences in the data of Riche and Bèrard, was the same, which these gentlemen prepared by means of paradibrombenzol.

Halogenized and Hydroxylized Organic Acids. E. ERLENMEYER and C. L. MULLER (Vol. XV. p. 49).

Combination in the Indigo Group. ADOLF BAEYER (Vol. XV. p. 50). The intermediate products between ortho-nitro-phenyl-propiolic acid and indigo are here studied by the author because he thinks, that the formation of indigo out of the first named product is due to an intermediate product formed, and by oxydation transformed into indigo.

Consequently ortho-dinitro-diphenyl-diacetylen was prepared, which, treated with fuming sulphuric acid gives rise to diisatogen,

a substance of remarkable interest. Of all artificially prepared substances it stands nearest to indigo and is easiest transformed into indigo; ammonic sulphydrate transforms it, cold, quantitatively into indigo.

$C_{16}H_8N_2O_4—2O + 2H=C_{16}H_{10}N_2O_2$.

The color is formed directly, without preliminary formation of indigo white or indoxyl, the diisatogen in contact with reducing agents becoming blue without either being dissolved or changing in form.

Syntheses by Means of Phenyl-Acetylene and its Derivatives. Adolf Baeyer and Ludwig Landsberg (Vol. XV. p. 57). Prepararation of diphenyl-diacetylen, and other derivatives.

Nicotinic Acid from Pyridin. Otto Fischer. (Vol. XV., p. 62.) Pyridin-sulpho-acid was transformed into cyanpyridin, which, treated with conc. hydrochloric acid, gave pyridin-monocarbonic acid, which latter is identical with nicotinic acid. A comparison of this acid and the nicotinic acid prepared from chinolin showed no difference whatever in properties.

Phoron from Glycerin. K. E. Schulze. (Vol. XV., p. 64.) Production of phoron from glycerin by fermentation.

Refraction and Heat of Combustion. Julius Thomsen. (Vol. XV., p. 66.)

Normal Cyanic Acid and Derivatives. E. Mulder. (Vol. XV., p. 69.) Preparation of ethyl ether of cyanic acid.

The Reducing Properties of Living Cells. J. Reinke. (Vol. XV., p. 107.) Controversial.

Contributions to the Chemistry of the Cerium Metals. B. Brauner. (Vol. XV., p. 109.) Studies in regard to the periodical system of the elements.

Synthesis of the so-called Alkyldisulphoxides. Robert Otto. (Vol. XV., p. 121.

Contributions to the History of Atropin. A. Ladenburg. (Vol. XV., p. 133.) Controversial.

On Determination of the Density of Gases. Heinrich Goldschmidt and Victor Meyer. (Vol. XV., p. 137.) Description of apparatus, &c.

ON THE ESTIMATION OF SULPHURETTED HYDROGEN IN GAS BY DIRECT WEIGHT.

By A. P. Hallock.

Last winter, while engaged in making some investigations in water gas, I made a few experiments in regard to the sulphur present, in order to see if I could not obtain some idea of in what form it was present. To do this I started to make some determinations of the total amount of sulphur in the gas. Using first the finished gas, which is the gas as it goes to the consumer, after being both enriched and purified, next the gas which had only been enriched but not purified, and lastly what is called the hydrogen. This is the water gas just as it comes from the gasogens, and before the addition of the naphtha vapor. It consists of a mixture of carbonic oxide, hydrogen, marsh gas, carbonic acid and sulphur compounds in varying proportions depending upon the kind of coal used.

With the finished gas I had but little trouble, for the total sulphur could be estimated in the same way as the sulphur in coal gas, by burning it in what is known as the Referee's apparatus. In this apparatus, the gas is burned very slowly at the lower end or mouth of a vertical trumpet-shaped chimney ; the upper end of the chimney having the small opening is connected with a condensing cylinder filled with glass beads in order to present the largest possible amount of surface. On a collar around the burner are placed some pieces of solid carbonate of ammonia, or a dish containing strong ammonic hydrate may be placed under the burner. Ammonia vapor is drawn up through the chimney by the current of air which supports combustion, and unites with the sulphurous acid to form a salt which condenses in the cylinder. After a little while the liquid condensed in the cylinder begins to trickle down the glass beads to the bottom, where it runs out through a small tube into a beaker or flask set underneath. After ten or more feet of gas have been burnt, the gas is turned off, the cylinder and beads washed, and the washings added to the contents of the beaker. After evaporating down to small bulk, the sulphur compounds are oxydized with bromine or chlorine to sulphuric acid, combined with barium, weighed as sulphate of barium, and from this the sulphur calculated. If the oxydation has been complete, the liquid dropping into the beaker will be colorless

or very nearly so, but when the sulphur has not been completely oxydized the liquid will have a decidedly yellow color.

With the finished gas the solution was always colorless or very nearly so, but when I attempted to estimate the sulphur in the unpurified gas, either before or after enriching, I always obtained a deeply-colored liquid, and the total amount of sulphur but little more than the amount obtained from the purified gas, and much less than I had reason to suppose existed. Why this should be, I am as yet unable to explain, but as it was actually the case, after a few repeated attempts I gave it up and started to make estimations of the amount of sulphuretted hydrogen, thinking if I could accomplish this it might give me some idea of the sulphur.

I tried making some determinations in an absorption tube for gas analysis, using binoxide of manganese as recommended by Prof. Bunsen, but I soon found that in cases like this where the quantity of sulphur was comparatively small and the amount of gas which could be used was also very small, the liability to error was so great that no satisfactory results could be obtained. I then concluded to try it by absorption in a U-tube in order to employ a large volume of gas. At first liquids were used, such as solution of acetate of lead, sulphate of copper, etc., but none of these worked well. At the suggestion of Mr. Lees, I tried dehydrated sulphate of copper, this absorbed the sulphuretted hydrogen very nicely, forming sulphide of copper and sulphuric acid; but the difficulty was that in order to completely dehydrate it, it was necessary to pulverize it, and in so doing, it generally became so finely powdered that it would pack in the U-tube and clog it up until the pressure would become sufficient to blow up through it, forming direct gas passages, through which the gas would pass and hardly be acted upon. About this time I was busily engaged with some other work and had no more time to devote to the sulphur determinations and left them for awhile. In the meantime, which was about the first of April, Mr. Lewis T. Wright, of England, published an article in the English *Gas Light Journal* on the estimation of sulphuretted hydrogen and carbonic acid in coal gas. In this article he mentioned the difficulty offered to the passage of the gas in the U-tube by the ordinary dehydrated sulphate of copper, and said he had overcome this by drying it first at about 100° C., then adding a little water to it and stirring it with a glass rod in order to slake it, which makes it granular, then drying it again at 100°C. and then at 155°C. At my first opportunity I prepared some in this manner and found it porous, and

it worked very nicely, completely overcoming the previous difficulty.

In order to give a better idea of the method employed, I will describe the apparatus which was used. It consisted first of a drying cylinder containing fused chloride of calcium, next to this was attached a weighed six-inch glass stoppered U tube containing the dehydrated sulphate of copper, a bit of cotton was put on the top of the sulphate in the U-tube to prevent any of it being blown out. To this U-tube was attached another weighed U-tube containing soda-lime and chloride of calcium to catch any carbonic acid and thereby enable me to determine it at the same time, and after this was placed, a meter to measure the quantity of gas passed through the tubes, and from the meter was a tube leading to a small gas burner where the gas was burnt.

The object of the first chloride of calcium was to dry the gas, for any moisture would be caught in the sulphate of copper tube and weighed as sulphuretted hydrogen. In burning the gas in the Referee's apparatus, I had to pass it through a wet meter first, to measure it, and in order to avoid a loss by the sulphur being washed out by the water, the water was replaced by glycerine and gas allowed to pass through it for a long time before using it, but in this case I was able to place the meter after my absorption apparatus, and thus avoid any error from this cause.

The apparatus was first tried on the pipe leading from the hydrogen main; after letting a sufficient quantity blow out to ensure that it was fresh gas, for when the gas was first turned on it failed to produce any stain on acetate of lead paper showing that the sulphur was being removed from the gas by the iron rust in the pipe. Sometime after it began to produce a stain, the apparatus was attached by means of a T-shaped pipe so that the gas could be kept burning all the time and thus have the flow of gas up the iron pipe much more rapid, and thereby have it as little acted upon as possible.

The gas was allowed to pass very slowly through the apparatus, not over one foot in three hours; after one foot had passed, the U-tube was detached and weighed, and found to have increased 0.198 grammes in weight, which would give 315.51 grains of sulphuretted hydrogen in 100 cubic feet, or 0.47 per cent. by volume. After this, four more determinations were made, allowing only one-half foot to pass each time, and although in this case the results had to be multiplied by 200 they agreed very closely, as may be seen by the following table. The column marked A gives the increase

in grammes of weight of the U-tube, B gives the number of grains of sulphuretted hydrogen in 100 cubic feet, and C the percentage :

	A.	B.	C.
No. 1.....	.1085	344.91	0.51 per cent.
No. 2.....	.1100	339.52	0.52 " "
No. 3.....	.1110	342.59	0.52 " "
No. 4.....	.1105	341.00	0.52 " "

These I consider as agreeing very closely.

A few days after I began the experiments upon the finished gas and was surprised to find that the U-tube gained more in weight than it did when using the unpurified, sometimes showing as much as 100 grains more in 100 cubic feet, while the Referee's apparatus, as used by Dr. Love and also by myself, did not give over 10 grains of total sulphur.

After a few attempts I found the sulphate of copper assumed a peculiar tint different from what I had seen while working on the unpurified gas, and, thinking that the sulphuric acid liberated might be in some way acting upon some of the hydrocarbons, I took some of the sulphate out of the tube and treated it with absolute alcohol and also with ether, and obtained a peculiar oily substance, apparently a mixture of hydrocarbons, but have not as yet been able to obtain a sufficient quantity of it to make any experiments upon it, but hope to be able to do so before long. This was sufficient to prove to me what caused the increase in weight of the U-tube and showed that it was not due to sulphuretted hydrogen.

I have not yet had an opportunity to try this method on unpurified coal gas, but according to Mr. Wright's statements it gives very satisfactory results.

ON THE FRACTIONAL DE-HYDRATION OF AMMONIUM ALUM, AND THE ATOMICITY OF ALUMINUM.

By J. W. Mallet.

In the course of experiments preparatory to my investigation of the atomic weight of aluminum,* I examined the behavior of crystallized ammonium-aluminum alum when dried under various conditions, among others when placed over sulphuric acid in a desiccator.

* Philosophical Transactions, 1880, Part III.

The question of the extent to which water of crystallization is removable, under well-defined conditions, from the alums, bears upon the adoption of the formula

$$M' M''' (SO_4)_2 \, 12 \, H_2O$$

or

$$M' M'''_2 (SO_4)_4 \, 24 \, H_2O$$

as correctly representing one of these salts. This question has been discussed by Lupton and Watts,† on the basis of the experiments of Hertwig;‡ Lupton§ and Heintz,‖ with the general result that in sundry cases the amounts of water lost, by heating to definite points of temperature, or by evaporation of a solution mixed with excess of sulphuric acid, point to the second of these formulæ, from which odd numbers—such as 19, 21 and 23—of molecules of water may be withdrawn, and which therefore cannot be halved and thus reduced to the simpler formula if but a single molecule of the anhydrous salt be supposed to be present in the residue.

Having ascertained that ammonium-aluminum alum loses largely of its water of crystallization by exposure to air of ordinary atmospheric temperature and pressure dried by a surface of sulphuric acid, and that the loss, even after it has become very slow, goes on for a very long time, I determined, as a separate question from that with which I was chiefly occupied, to continue the drying under these conditions until loss of weight should cease, and see what number of molecules of water would be left. The experiment lasted more than *two years*, weighings being made at first at intervals of a few hours, then of a day, of two days, of two weeks, and, toward the end, of a month. The temperature of the room, which in winter was warmed continuously by night as well as day, ranged generally about from 20° to 27° C., but occasionally passed these limits to a considerable extent during long-continued periods of hotter or colder weather out of doors, and such variations of temperature as occurred made themselves perceptible in the more or less rapid loss of weight of the salt experimented on. The alum was in the form of a fine crystalline meal, spread out to the depth of 7 or 8 millimeters over the surface of a platinum capsule about

† Watts' Dictionary of Chemistry, 3d Suppl., Part I., pp. 65, 66.

‡ Pogg. Ann., lv., 99.

§ Chem. Soc. Jour. (Lond.) [2], xiii., 201.

‖ Pogg. Ann., iv., 331.

10 cm. in diameter. The desiccator was of large size, and furnished with a drying tube, containing sulphuric acid, communicating with the outside air, so that the pressure within scarcely differed at any time from that of the atmosphere without.

The following were the results obtained.

Weight of alum at commencement of experiment=39.1654 grams.

Loss after	0	day	9½	hours——	.0839	gram.	
" "	1	"	2	" ——	.2419	"	
" "	1	"	9	" ——	.3261	"	
" "	2	"	0	" ——	.4423	"	
" "	3	"	2	" ——	.6944	"	
					(.7781	"	=1 mol. H_2O.)
" "	4	"	4	" ——	.8962	"	
" "	5	"	8½	" ——	1.1703	"	
					(1.5562	"	=2 H_2O.)
" "	7	"	0	" ——	1.6027	"	
" "	9	"	0	" ——	2.1123	"	
					(2.3344	"	=3 H_2O.)
" "	11	"	0	" ——	2.5218	"	
" "	13	"	0	" ——	2.9480	"	
					(3.1125	"	=4 H_2O.)
" "	15	"	4	" ——	3.3821	"	
" "	17	"	0	" ——	3.7365	"	
					(3.8906	"	=5 H_2O.)
" "	19	"	0	" ——	4.1056	"	
" "	21	"	0	" ——	4.3985	"	
					(4.6687	"	=6 H_2O.)
" "	23	"	0	" ——	4.6811	"	
" "	25	"	0	" ——	4.8855	"	
" "	27	"	0	" ——	5.1411	"	
" "	29	"	0	" ——	5.3849	"	
					(5.4468	"	=7 H_2O.)
" "	31	"	0	" ——	5.7015	"	
" "	33	"	0	" ——	6.0237	"	
					(6.2250	"	=8 H_2O.)
" "	35	"	0	" ——	6.3069	"	
" "	37	"	0	" ——	6.5191	"	
" "	39	"	2	" ——	6.7106	"	
					(7.0031	"	=9 H_2O.)

Loss after 42	day	2	hours	——	7.0279	gram.
					(7.8812	" =10 H_2O.)
"	" 57	" 23	"	——	8.2636	".....
					(8.5593	" =11 H_2O.)
"	" 73	" 1	"	——	9.1001	"
					(9.3374	" =12 H_2O.)
"	" 103	" 3	"	——	9.8668	"
					(10.1156	" =13 H_2O.)
"	" 133	" 6	"	——	10.6028	"
					(10.8937	" =14 H_2O.)
"	" 168	" 7	"	——	10.9848	"
"	" 198	" 23	"	——	11.5513	"
					(11.6718	" =15 H_2O.)
"	" 229	" 23	"	——	12.2015	"
					(12.4499	" =16 H_2O.)
"	" 266	" 0	"	——	12.7269	"
					(13.2380	" =17 H_2O.)
"	" 298	" 0	"	——	13.4779	"
					(14.0062	" =18 H_2O.)
"	" 330	" 22	"	——	14.0662	"
"	" 364	" 1	"	——	14.4133	"
"	" 395	" 6	"	——	14.5237	" [Put fresh acid into desiccator.
"	" 425	" 23	"	——	14.5670	"
"	' 467	" 3	"	——	14.5955	"
"	" 487	" 3	"	——	14.6146	"
"	" 519	" 3	"	——	14.6341	"
"	" 549	" 5	"	——	14.6528	"
"	" 577	" 3	"	——	14.6675	"
"	" 608	" 6	"	——	14.6793	"
"	" 639	" 0	"	——	14.6933	"
"	" 669	" 6	"	——	14.7277	"
"	" 705	" 1	"	——	14.7595	"
					(14.7843	" =19 H_2O.)
"	" 730	" 2	"	——	14.7847	"
"	" 761	" 3	"	——	14.7875	"
"	" 800	" 0	"	——	14.7875	"
"	" 811	" 3	"	——	14.7858	"
"	" 998	" 23	"	——	14.7955	"

Hence it appears that by exposure to dried air under the conditions stated, ammonium alum loses 19 out of 24 molecules of

water, and thus adds one case of definite partial de-hydration to those cited by Lupton and Watts * in favor of the layer formula ——the observed residues standing as follows (Watts' table, copied from Lupton's paper, with the case now reported added in brackets):

$(NH_4)_2Al_2(SO_4)_4 . H_2O$	$K_2Al_2(SO_4)_4 . H_2O$	$(NH_4)_2Fe_2(SO_4)_4 . H_2O$	$K_2Fe_2(SO_4)_4 . H_2O$
	$K_2Al_2(SO_4)_4 . 3H_2O$		$K_2Al_2(SO_4)_4 . 3H_2O$
$\left((NH_4)_2Al_2(SO_4)_4 . 5H_2O\right)$	$K_2Al_2(SO_4)_4 . 5H_2O$		

If the rate of loss in the interval between the weighings next above and below that corresponding to each molecule be assumed as uniform, it appears that the

1st	molecule	of	water	was lost in	85 hours
2d	"	"	"		$78\frac{1}{2}$ "
3d	"	"	"		$78\frac{1}{2}$ "
4th	"	"	"		90 "
5th	"	"	"		96 "
6th	"	"	"		122 "
7th	"	"	"		155 "
8th	"	"	"		120 "
9th	"	"	"		182 "
10th	"	"	"		231 "
11th	"	"	"		282 "
12th	"	"	"		459 "
13th	"	"	"		744 "
14th	"	"	"		1121 "
15th	"	"	"		1065 "
16th	"	"	"		1024 "
17th	"	"	"		969 "
18th	"	"	"		964 "
19th	"	"	"		9646 "

Taking into account the changes of atmospheric temperature, especially the more persistent changes due to season, during the time occupied by the experiment, which began on the 3d of August, there seems to have been a gradual decrease in the rate of loss throughout, unmarked by any abrupt variations, except as regards the last molecule of water given off, the removal of which took

* Loc. cit.

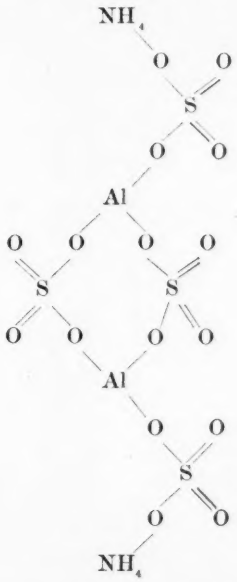
more than a year, and more than as long as all of the 18 preceding molecules.

While this experiment sustains the conclusion of Watts that the proper formula for the alums is:

$$M^{I}_{2}M^{III}_{2}(SO_4)_4 . 24\ H_2O$$

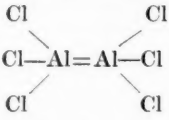
I do not perceive that the further conclusion at which he arrives is valid—viz. "*This being admitted, it follows that aluminum, chromium and iron must be tetratomic.*" There is no direct evidence of aluminum exhibiting tetrad character.

All its general relations are those of a triad element. In the alums it is not necessary to assume any "linking function" on the part of the two atoms of aluminum. The constitution of ammonium-aluminum alum, for instance, may be:



If, however, it be assumed that the two aluminum atoms are directly combined, either in this salt or in the haloid compounds of the metal it seems more probable that in such cases there is double linking, and that aluminum is to be considered as triad or *sometimes pentad.* Thus the chloride, taking the for-

mula Al_2Cl_6 as established by Deville and Troost's determination of the vapor density, may be represented as :



This view seems to reconcile the otherwise conflicting evidence as to atomicity derived from the observed vapor densities of aluminum chloride on the one hand and aluminum methid and ethid on the other. The determinations of Buckton and Odling for the vapor densities of the latter of these compounds are quite inconsistent with the common assumption that aluminum is tetrad.

UNIV. OF VIRGINIA, *Sept.* 20, 1882.

ON NITROSACCHAROSE : A CORRECTION.

BY ARTHUR H. ELLIOTT, PH. B., F. C. S.

In Vol. IV, p. 147 of this Journal, will be found a paper by me on nitrosaccharose, and at the end of said paper some theoretical conclusions are given which I desire to withdraw.

When the paper was written I was about starting on my summer vacation, and I did not see it again until after it was printed, and a second reading of it showed me I had made a mistake in my figures.

Prof. C. F. Chandler also kindly called my attention to the improbability of the formula given by me to nitrosaccharose. But in assigning the formula given, I was led into error by a corresponding formula found in the English edition of Wislicenus Strecker's handbook, where the octacetate of saccharose is given as $C_{12}H_{14}(C_2H_3O_2)_8O_{11}$ the correct formula being $C_{12}H_{14}(O.C_2H_3O)_8O_3$ eight molecules of the group C_2H_3O replacing the hydrogen in eight molecules of hydroxyl ; thus giving saccharose the formula $C_6H_7(OH)_4O—O—O(OH)_4C_6H_7$

In regard to the error in my figures, I took the percentage of of cane sugur found and added it to the percentage of NO_3. Correctly, I should have taken the percentage of cane sugar minus the hydroxyl, and added this residue ($C_{12}H_{14}O_3$) to the NO_3 found. By

doing this 28.55, the percentage of cane sugar found is equal to 17.19 per cent. of $C_{12}H_{14}O_3$; which added to the NO_3 found (41.45) gives a total percentage of 58.64; and the ratio of cane sugar to NO_3 becomes 1 to 8.028 instead of 1 to 8.009, as given before. Practically the ratio of the molecules of cane sugar and NO_3 remain the same, 1 to 8.

From a consideration of this relation and of the formula for saccharose quoted above, the true formula for nitrosaccharose becomes $C_{12}H_{14}(O.NO_2)_8O_3$ or an octonitrate; thus coming into the same class of organic nitrates as nitrocellulose and nitroglycerine.

SCHOOL OF MINES, *Sept.* 1882.

REVIEW OF PAPERS ON ANALYTICAL CHEMISTRY (PROXIMATE) WHICH HAVE APPEARED DURING THE YEAR 1882.

By E. Waller, Ph. D.

Milk Analysis.—To determine Water, *Marpmann* (*Biedermann's Centrlb. fur Agrikult. Chem. XI., pt.* 2) proposes to fill a calcium chloride tube with cotton wool, then to saturate the cotton with the milk, and to dry by drawing a stream of hot dry air through the tube. The fat may also be determined in the same sample by extraction with benzol or ether.

O. Hehner gives the results of experiments on the methods for milk analysis adopted by the Soc. of Pub. Anal. (*Analyst*, VII., 60). In determining water the operation was more rapid on the water bath than in an oven. After drying for three hours an amount varying from 0.06 to 0.29 % may be driven out by further heating. A temperature above 100° C. apparently causes decomposition and loss of solids not fat. For butter fat the Soxhlet apparatus removes about 0.2 % more than boiling out with three or more successive quantities of ether. Nothing is gained by granulating the milk residue, by allowing the ether to act for a long time, or by adding 10 % absolute alcohol to it. Portions of the ash are volatile at red heat.

A. Mayer states that the oil from artificial butter has a much lower specific gravity than that from natural butter. (*Fuehling's Landw. Ztg.* XXXI., 92)

The amount of copper normally present in wheat is stated by *J. van D. Berghe* as 0.008 to 0.01 % (*Les Mondes*, I., [3] 209).

To test bread for the presence of alum, *A. W. Blyth* (*Analyst*, VII., 16) proposes the use of slips of gelatine. The bread is crumbled into a vessel, a couple of slips of dry commercial gelatine placed with it, and distilled water added sufficient to cover the materials. On standing over night, the alum concentrates in the gelatine, and when dipped in fresh tincture of logwood, to which an equal bulk of 10 % solution of ammonium carbonate has been added the slips will dye blue if alum was present. When alum is not present, the slip becomes reddish brown, the tint being removable by soaking in glycerin. Experiments upon alumed bread seemed to show that much of the alum added remains as such after baking. Nearly all, if not quite all, of the alumina added as alum may be removed from the bread by soaking it in a 5 % HCl. solution.

In EXAMINING TEA, *J. Carter Bell* (*Analyst*, VII., 7) gives the results of the examination of 58 samples. The variations were as follows :

Per cent. of ash.....	5.64 to 8.58
Per cent. of ash soluble in water.....	32.70 to 67.41
Per cent. alkalinity of ash ($=K_2O$).....	14.24 to 30.57

In EXAMINING ROASTED COFFEE, *A. Smetham* (*Analyst*, VII., 73) concludes from the examination of seven genuine samples, first, that moisture (loss at 100° C.) should always be first determined. The per cent. of "crude fibre" is the best guide where chicory is the adulterant. The determination of oil, cellulose, ash (soluble and insoluble), nitrogen, and sp. gr. of the 10 % infusion, are principally of service as checks on the work.

The crude fibre was found to vary between 73.71 and 75.7 %, referred to the dry samples. It was determined by boiling 2 gms. of the samples with three successive lots of water (100 cc. each), and finally washing on a tared filter until the washings were colorless.

For COLORIMETRIC ESTIMATION OF SALICYLIC ACID in articles of food, urine, &c., *MM. Pellet and De Grobert* (*Revue des Industries*, No. 53, 1882) propose ferric chloride solution (sp. gr. 1.005 to 1.008) containing 0.5 gms. free acid per 100 cc. In testing butter by this method, 25 gms. are shaken with 5 or 6 drops sulphuric acid (of 30° B.) and 75 cc. benzol. The total volume is then made up to some convenient bulk (100 cc.) It is then filtered, and 5 or 10 cc. mixed with the same bulk of water, and a few drops of the ferric chloride solution added. The mixture is gently shaken, the production of an emulsion being avoided, and the tint compared with that obtained in a standard solution.

EXAMINATION OF WINES. General methods are given by *J. Nessler and M. Barth* (*Fres. Zeitz. Anal. Chem.* XXI., 43) and *L. Roesler* (*Chem. Zeit.*, VI., 306).

For EXTRACT, the first named authors find that evaporation at 100° C. for three hours does not remove the glycerin. Some loss by decomposition of non-volatile acids will be experienced, unless a known bulk of Ba $(HO)_2$ solution is added. For genuine wines, the extract less acids is rarely under 10 %. *Roesler* asserts that evaporation at temperatures above 80° causes both loss of glycerin and decomposition of the residue. He recommends evaporation at 60° C. in partial vacuum.

For added SUGAR *Nessler and Barth* prefer a modification of Neubauer's polariscope test. Cane sugar if present is partially or wholly inverted by the acids of the wine on evaporating. Caramel has no optical effect. *Roesler* decolorizes with bone black and uses the Fehling test.

For FREE TARTARIC ACID *Nessler and Barth* evaporate a portion, add alcohol until no further precipitation ensues, filter, evaporate off the alcohol, add water, clarify by bone black, and then add one or two cc. of 20% solution of calcium acetate. The amount of free tartaric acid should not be over one-sixth the amount of the non-volatile acids.

Roesler first determines tartar by adding 50 cc. ether to 10 cc. of wine, allowing to stand 24 hours, washing with ether, dissolving in boiling water and titrating. Then 10 cc. of the wine is neutralized with standard KOH solution, 40 cc. of the wine added, and 10 cc. of the mixture treated as above. The excess of the acidity in the second case is due to free tartaric acid.

Nessler and Barth also state that the ASH should be at most 1.4%. In some cases the wines may have been fraudulently diluted and the percentage of ash increased by use of SALT. In such a case the ash does not readily burn white. To detect this fraud, the wine is decolorized by bone black and Volhard's method used. The usual amount of Cl in wines is under 0.002%, the maximum 0.005%.

The last named authors also state that the maximum amount of CITRIC ACID in genuine wines is 0.002 to 0.003 per cent., but that acid is frequently added as such or in tamarinds (which contain 13.5%.

To determine it, 100 cc. of the wine are evaporated to 7 cc., cooled, the soluble portion extracted by alcohol (of 80%) the residue diluted to 20 cc., *partly* neutralized with milk of lime, (for red wines bone black is also added at this point) filtered, diluted to 100 cc., and the citric and tartaric acids separated by about one cc. of neutral lead acetate. From this combination the lead is separated by agitation with H_2S water, and filtering. After removing the H_2S, lime is added to render the solution alkaline, and after filtering it is made slightly acid with acetic acid and allowed to stand to separate the calcium tartrate. In the filtrate the acetic acid is expelled by heat and the solution is finally boiled until the calcium citrate separates in crystalline form when it is filtered and weighed. *Roesler* determines the GRAVITY preferably by Sprengel's pyknometer; ALCOHOL by distilling off two-thirds, making the distillate up to the

original volume and taking the gravity ; TANNIC ACID by distilling off the alcohol, making up to the original volume, agitating with bone black, filtering, and applying Neubauer's modification of Lœwenthal's method, (permanganate and indigo carmine); ACETIC ACID by adding baryta hydrate, distilling off alcohol, and then distilling with phosphoric acid (Kessel-Neubauer's method); GLYCERIN by evaporating (100 cc.) down to one-third, rendering slightly alkaline with slaked lime, evaporating to dryness, extracting with alcohol, evaporating this again to dryness and extracting the glycerin from the residue by a mixture of alcohol and ether, (Neubauer-Reichardt's method); and NITROGENOUS CONSTITUENTS by evaporating 10 to 20 cc. in a thin glass vessel, pulverizing residue glass and all, and making a combustion as usual.

For TANNIN AND ŒNOGALLIC ACID, *F. Jean* (*Comtes Rend.* XCIV. No. 11) evaporates 50 to 100 cc. down to small bulk—adds dry precipitated silica, dries at 60 to 70°C., extracts with ether containing a little hydrochloric acid, evaporates the ethereal solution, and dissolves in water, up to the original bulk. In 10 cc. of this solution the combined acids are determined by titrating with iodine, and in the remainder the œnogallic acid is determined (after shaking with rasped hide to remove tannin) by titration again with iodine.

For GLYCERINE in sweet wines *E. Borgmann* (*Fres. Zeits. Anal. Chem.*, XXI, 239) evaporates 100 cc. to a syrup, extracts the residue with 100 cc. of absolute alcohol, and then adds 150 cc. of ether. The sugar settles to the bottom, leaving the glycerine in solution.

For RED COLORING MATTERS *J. De Groot* (*Repert der Analyt. Chem*, II, 95) adds to a wine or other liquid to be tested, one volume chloroform and three volumes absolute alcohol, and agitates briskly; three volumes of distilled water are then carefully added without shaking. Many coloring matters separate at the point where the two liquids are in contact. A few, as, for instance, fuchsin, pass into the chloroform. By the manner of separation or the coloration imparted to one or both liquids, many of the coloring matters may be identified. To detect BORDEAUX RED IN WINES *C. Thomas* (*Chem. Zeit.*, VI, 244) precipitates the wine with barium hydrate—filters—removes the excess of barium by ammonium carbonate, and in the filtrate by evaporation, ignition, etc.; tests for the presence of sulphuric acid arising from the presence of a sulpho compound. *M. Guichard* (*Journ. de Pharm. et de Chim.*, May 1882) tests for the same coloring matter by dyeing silk with the wine. The natural coloring matter gives only a violet or lilac

shade, which turns greenish with ammonia, while the Bordeaux red dyes the silk a garnet shade, turning darker with ammonia; also a drop of genuine wine let fall on a lump of chalk moistened with alum solution gives a violet gray spot; whereas, if Bordeaux red is present, a red stain appears.

F. Musculus and *C. Amthor* (*Repert. Analyt. Chem.*, II, 142) find that the natural WINES OF A BAD YEAR contain but little alcohol and much acid, but contain more ash, extractive and phosphoric acid than even the best wines of a good year.

For SULPHUROUS ACID IN WINES *B. Haas* (*Ber. D. Chem. Ges.*, XV, 154) distils off about half of a measured quantity in a current of CO_2 into a bulb tube containing iodine solution, when the sulphuric acid formed may be determined by $BaCl_2$. *L. Lieberman* (*Ber. D. Chem. Ges.*, XV, 439) distils in a similar manner, oxidizing by the use of HNO_3. The last-named author makes a qualitative test upon the distillate by using iodic acid and chloroform. A small amount of SO_2 gives the violet coloration to the chloroform; larger amounts a yellowish brown.

OILS, &C.—To distinguish between OLIVE AND COTTON SEED OIL *M. Zecchim* (*Les Mondes*, May 13, 1882) agitates 5 cc. of the oil with 10 cc. of colorless nitric acid (sp. gr. 1.4), and then allows it to stand for five or six minutes. Pure olive oil will then appear a light ash gray color, with a slight yellow reflex; after some time only it becomes dark colored. Cottonseed oil becomes golden yellow, soon turning coffee brown. Mixtures of the two give intermediate shades.

To detect ROSIN OR PARAFFIN OIL IN LARD, TALLOW, ETC., *W. Rodiger* (*Chem. Zeit.*, 1882, 118) saponifies and then extracts with benzine. If much soda lye is necessary to cause the separation of the soap from its solution in water, palm or cocoanut oil is probably present.

To determine ROSIN IN FATS, *T. S. Gladding* (*Am. Chem. Jour.*, III, 416), dissolves about 0.5 gm. in 20 cc. alcohol (95 %) and saponifies by alcoholic potash solution, the addition being controlled by the indication of a drop of phenol phthalein in the solution. A slight excess of potash is used. After insuring saponification by heating, the solution is cooled, and then transferred to a 100 cc. cylinder by the use of ether, enough being used to bring the volume up to 100 cc. After thorough mixture by agitation, 1 gm. pure pulverized $AgNO_3$ is added, and the mixture again well agitated for 10 or 15 minutes. The silver resinate goes into solution in the ether. An

aliquot portion of the clear ether solution is then siphoned off into another cylinder, and agitated with 20cc. of HCl. (1:2 by vol.) to set the rosin free. On evaporating a measured amount of the last ether solution the rosin is left behind, together with a little oleic acid (0.00235 gm. to every 10 cc. of ether solution taken.)

Ulex tests for FOREIGN SEEDS IN RAPE-SEED CAKE (*Repert. Anal. Chem.*, 1882, p. 3,) by distilling with water, and testing for sulphur in the distillate by oxidation with bromine and precipitation with barium salt, 233 parts Ba SO_4 = 99 parts oil of mustard. Rape-seed cake contains only 0.0238 % of that oil.

ALKALOIDS.—A solution of 1 gm. melted zinc chloride in 30 cc. of water is proposed by *Czumpelitz* (*Monit. Sci.* Feb. 1882). The substance to be examined is first dried, then moistened with the reagent and dried again. The colors produced are:

Strychnine,	Scarlet,	Quinine,	Pale Yellow,
Thebaine,	Yellow,	Digitaline,	Maroon,
Narceine,	Olive green,	Salicine,	Violet red,
Delphinine,	Red brown,	Santonine,	Violet blue,
Berberine,	Yellow,	Cubebine,	Purple,
Veratrine,	Red.		

When both strychnine and brucine are present the color obtained is a dirty yellow.

R. C. Woodcock (*Chem. News* XLV, 79) proposes TO SEPARATE ALKALOIDS from articles of food, &c., by the use of cubes of jelly prepared from a 6 % solution of gelatine. (Applied by *Dupre Analyst*, I, 186, to separate certain adulterants of wines.) In an experiment with strychnine, one part was satisfactorily separated from a million parts of substance.

H. Beckhurst (*Chem. Centr. bl.* XIII, 258) finds that the reduction of ferric compounds is not characteristic for the PTOMAINES. To extract alkaloids from CINCHONA BARK *J. E. De Vrij*, (*Jour. Pharm. Chim.*, May, 1882) recommends Prollius's mixture consisting of:

Ether	88 parts.
Ammonia	4 parts.
Alcohol (92 to 96 %)	8 parts.

For the extraction and determination of the alkaloids in the above barks, methods are also given by *M. Fluckiger* (*Jour. Pharm. Chim.* Feb. 1882) and *E. R. Squibb* (*Ephemeris* I, 76 and 105). The first named boils the powdered bark with four times its weight of water

and then adds milk of lime, after which the mixture is evaporated to dryness. The dry mass is exhausted with ether, and after the addition of a little hydrochloric acid (36 cc. of $\frac{1}{10}$ normal HCl. to the extract from 20 gms. of bark), the ether is distilled off. The solution is then acidified with HCl. and filtered. The cold solution is partly neutralized by $\frac{1}{10}$ normal NaHO solution 40 cc., allowed to stand until it clears, decanted or filtered if necessary, and soda lye (of sp. gr. 1.3) added to complete precipitation of the bases. The precipitate is dried between filter paper, then over sulphuric acid, and finally in an air bath at 100° C. before weighing. To separate the quinine the precipitate is boiled with thirty times its weight of water for an hour, and filtered boiling. On cooling, the quinine hydrate is deposited. If pure, the dry alkaloid is soluble in twenty times its weight of ether. *E. R. Squibb* takes 1.25 gm. quick lime, adds 30 cc. hot water, and as soon as it is slaked stirs in 5 gms. pulverized bark, and allows it to digest for some time. After drying over a water bath, the powder is digested in a closed flask over a water bath with 25 cc. amylic alcohol for four hours; 60 cc. of ether are then added and the digestion continued for an hour more with frequent shaking. It is filtered and washed with a mixture of 10 vols. amylic alcohol to 40 of ether. The ether is then boiled off, and the residue transferred to a small flask by the use of amylic alcohol. The solution and washings should not be over 10 cc. Then 6 cc. water and 4 cc. normal oxalic acid solution are added, and the mixture shaken. The aqueous solution is then filtered off and the amylic alcohol washed repeatedly with diluted oxalic acid solution. The solution is evaporated to about 15 cc., 20 cc. pure chloroform added, and a slight excess of normal soda solution. The alkaloids are thus separated, and are taken up by the chloroform, which, after washing, may be evaporated, leaving the alkaloids pure.

By shaking the alkaloids thus obtained with 5 gms. ground glass and 5 cc. ether, and pouring the mixture upon a filter wetted with ether, rinsing the adherent particles on to the filter until 10 cc. have passed through, and then washing with ether until a second 10 cc. (caught separately) have passed through, and evaporating these portions of ether separately, a basis for the estimation of the quinia is obtained. The first 10 cc. contains quinia plus some other alkaloids, the second 10 cc. approximately the same amount of alkaloids, not quinia, as the first. The difference between the weights of the residues left is therefore quinia.

E. R. SQUIBB also gives a process for the ASSAY OF OPIUM (Ephe-

meris I, 14), a modification of Fluckiger's process. Careful sampling and the determination of moisture are both indispensable. 10 gms. of the sample are first digested with 100 cc. water with frequent shaking for several hours. It is then filtered and percolated with water until the filtrate has a bulk of 120 cc. The residue is again shaken with more water and the percolation repeated, the filtrate being kept separate, and evaporated down to about 20 cc. before adding the stronger solution (of 120 cc.), the object being to avoid the effect of prolonged heat upon the major part of the alkaloids. The residue should be dried at 100° C. and weighed. The entire extract is finally evaporated to 20 cc.—cooled—5 cc. of alcohol added and well stirred in, then transferred to a tared flask, being mixed in with about 5 cc. water, 5 cc. more alcohol is added and after thorough admixture 40 cc. of ether. After vigorous shaking 4 cc. of ammonia (sp. gr. 0.96) is added, and the shaking is repeated until the crystals of morphia begin to separate, when the flask must be put aside for 12 hours. The ether solution is then decanted off through a filter wetted with ether, 20 cc. of ether is added to the flask and rinsed around without shaking and decanted through the filter. The funnel should be kept covered as much as possible while the ether is filtering through. Finally pour the aqueous solution on to the filter, rinse with a little water. Dry the filter and contents first with bibulous paper, afterward in the air bath (at 100°C) weigh, and deduct weight of filter. Invert the flask, dry and weigh it to get the weight of the morphia crystals which may adhere. The crystals of morphia are light brown or a loss has been sustained. 0.1 gm. of the precipitate when pulverized should dissolve completely on shaking in a corked test tube with 10 cc. of lime water (absence of narcotine). A description and criticism of the various methods for determining NICOTINE IN TOBACCO are given by *R. Kissling* (*Fres. Zeits. Anal. Chem.*, XXI. 64). The conclusion reached is that only Schloesing and Dragendorff's methods are at all satisfactory. The results of a number of experiments are detailed, and a list of analyses of 36 tobaccos given.

For the ESTIMATION OF TANNIN *F. Simaud* (*Dingl. Jour.*, CCXLIV., 391) proposes a modification of Lœwenthal's method. In extracting, paper saturated with a solution containing ferrous chloride and sodium acetate is used to determine when the extraction is complete. 10 cc. of the extract should not require over 12 cc. of the permanganate solution. The permanganate solution should be one gm. per litre, 1 cc.=0.00135 tannin, 20 cc. of the indigo solution

should be equivalent to 18 to 20 cc. of permanganate. 20 cc. of the solution is used for each determination. The gelatine solution is of the strength, &c., prescribed by Lœwenthal. To obtain satisfactory results close attention must be paid to the degree of dilution and the amount of acid used.

To DETERMINE GLYCERINE in dilutions thereof, it is proposed, (*Jour. Soc. Chem. Ind.*, I, 75), to moisten 5 to 60 gms. PbO_2 with 2 to 3 gms. of the dilution, and to dry the mixture for six hours in a desiccator, then one hour at 100° C. and finally one hour at 120° C. when it is cooled and weighed. $C_3H_6O_3Pb$ forms. The gain in weight multiplied by 1.3429 gives the weight of glycerine actually present.

To DETERMINE PHENOL *P Diacosa* (*Repert. Anal Chem.*, II., 137), proposes a reversal of the usual volumetric methods, viz., 20 cc. of bromine water the strength of which has been determined by the use of a standard solution of phenol, receives the addition of the solution containing phenol until the clear solution no longer acts upon iodized starch paper.

Jorissen's REACTION FOR FUSEL OIL.—A red coloration with anilin and H.Cl. is stated by *K. Forster* (*Ber. D. Chem. Ges.*, XV, 230), to be due to the presence of furfurol and not to that of amyl alcohol.

To determine ACETON IN WOOD SPIRIT *M. Kramer* (*Monit. Sci.*, Jan., 1882), adds 1 cc. of the sample, first 10 cc. of binormal solution of soda, then 5 cc. of binormal solution of iodine and agitates. The precipitate of iodoform is dissolved in 10 cc. of ether, and 5 cc. are evaporated on a weighed watch glass. From this the proportion of acetone is calculated. If the wood spirit is to be used in the manufacture of coal tar colors, not over 1 per cent. of aceton should be present.

To TEST ASPHALTUM, *D. Claye* (*Jour. Soc. Chem. Ind.* I., 203), dissolves in CS_2, filters, and evaporates off the solvent. The residue is then pulverized, and 0. 1gm. is treated with 5 cc. fuming sulphuric acid for 24 hours, and then poured slowly into 10 cc. of water with continuous stirring. Pure asphalt gives a clear light yellow solution, while the adulterants—pitch, coal tar, etc.—give a dark color.

URINE EXAMINATIONS—FOR UREA, *J. R. Duggan* (*Am. Chem. Jour.*, IV, 47), obtains the best results by first mixing the urea and soda solutions and then adding the bromine. The results of *T. G. Wormley's* experiments with Apjohn's apparatus (*Chem. News*, XXI, 37), are given as follows (*Chem. News*, XLV, 27): To

insure the elimination of the whole of the nitrogen—1. The reagent must be freshly prepared. 2. The urea solution should be wholly added to the reagent, none of the latter being allowed to mix with the urea solution in the containing tube. 3. The amount of urea operated upon should not exceed one part to 1200 of the diluted reagent.

The volumetric uranium solution for PHOSPHORIC ACID cannot, according to *G. Guerin* (*Jour. Pharm. Chem.*, Feb., 1882), be successfully applied to urine examinations. According to this author, the uranium solution only gives accurate results when standardized in the presence of the same kind of salts as those existing in the substance to be analyzed.

List of books published on analysis during 1882 :

THE LABORATORY GUIDE: A Manual of Practical Chemistry, etc. By *A. H. Church.* Fifth Edition. London: J. Van Voorst.

TABLES FOR QUALITATIVE CHEMICAL ANALYSIS. *H. Will.* Third American Edition. Edited by *C. F. Himes.* Philadelphia : H. C. Baird & Co.

HANDBOOK OF VOLUMETRIC ANALYSIS. *F. Sutton.* Fourth Edition. London: J. and A. Churchill.

COMMERCIAL ORGANIC ANALYSIS. Vol. II. *A. H. Allen.* London : J. and A. Churchill.

QUANTITATIVE ANALYSE AUF ELEKTROLYTISCHEM WEGE. *Dr. Alexander Classen.* Aachen : J. A. Mayer.

ANALYSE VON PFLANZEN UND PFLANZEN-THEILEN. *Dr. Georg Dragendorff.* Gottingen : Vandenhoeck & Ruprecht.

FOODS : THEIR COMPOSITION AND ANALYSIS. *A. W. Blyth.* London : Charles Griffin & Co.

MANUAL OF SUGAR ANALYSIS. *J. H. Tucker.* New York : D. Van Nostrand.

HANDBOOK OF THE POLARISCOPE. From the German of Landolt. By *D. C. Robb* and *V. H. Veley.* London : MacMillan & Co.

ABSTRACTS.

Abstracts from the *Journal of Chemical Society*, London, by Arthur H. Elliott, Ph. B., F. C. S.

On the Action of Aldehydes on Phenanthraquinone in Presence of Ammonia. (Third Notice). By Francis R. Japp, M. A., Ph. D., and Fred W. Streatfield. Vol. XL., p. 146.

This paper is a continuation of the work of the former of the two authors and Mr. Wilcock (*Chem. Soc. J. Trans.*, 1881, p. 228). The authors give an account of the product obtained by the action of salicylaldehyde upon phenanthroquinone. It has the composition $C_{21}H_{14}N_2O$; crystallizes in very fine silky needles; fuses and decomposes at 270—276°C.; its best solvents are glacial acetic acid and amyl alcohol. Behaves like a phenol with caustic alkalies forming compounds. It has the properties of a weak base also. Fusion with potash gives salicylic acid.

A benzoyl derivative of the above compound was formed, which consisted of crystals of two forms, cubes and needles, fusing at 218—220°C. Analysis led to the formula of a mono-benzoyl derivative $C_{21}H_{13}(C_7H_5O)N_2O$. Authors discuss the constitution of the original $C_{21}H_{14}N_2O$.

The action of parahydroxybenzaldehyde upon phenanthraquinone as above is also described. The product is obtained in fine white needles, not fusing within range of mercury thermometer. Soluble in glacial acetic acid and amyl alcohol, also in dilute caustic alkalies. Its formula is given as $C_{21}H_{14}N_2O$. An acetyl derivate of the preceding body was formed. It crystallizes in needles which fuse at 205—210°C. The formula is $C_{21}H_{12}(C_2H_3O)N_2O$; it is very soluble in glacial acetic acid.

Phenanthraquinone as above was acted upon by orthomethoxybenzaldehyde. The purified product was yellow needles fusing at 207—208.5°C. The mother liquor gives white crystals, fusing at 144.5—145.5°C. The yellow product gave formula $C_{22}H_{16}N_2O$; it is readily soluble in boiling benzine, amyl alchol, and glacial acetic acid, much less soluble in the cold; it is insoluble in dilute acids and caustic alkalies. The white product gave the formula $C_{22}H_{15}NO_2$; it is readily soluble in hot benzine, amyl alcohol and glacial acetic acid; insoluble in dilute acids and caustic alkalies.

Application of the Aldehyde and Ammonia Reaction in Determining the Constitution of Quinones. By Francis R. Japp, M.A., Ph.D., and Fred. W. Streatfield. Vol. XL., p. 157.

Authors apply the reaction to chryso-quinone and beta-naphtha-quinone. The chryso compound was obtained in silky needles, fusing at 259–265° C., and of a slight yellow tinge. Analysis gave $C_{25}H_{15}NO$ as formula. Sublimation gave a white product. The new body is *benzenylamidochrysole*.

The experiment with beta-naphtha-quinone gave negative results.

On the Action of Sodium Hydrate and Carbonate on Felspars and Wollastonite. By Walter Flight, D.Sc., F.G.S. Vol. XL, p. 159.

Author experimented upon adularia from St. Gothard, microlin from Siberia, albite from the Dauphiné, and a specimen of wollastonite from Sweden. These were heated in fine powder with solutions of sodium hydrate of different strengths, and also with solution of sodic carbonate. In the case of sodium hydrate, microlin lost 14.693 per cent. in strong solution and 7.453 in weak solution. Albite, under the same treatment, lost 23.1 per cent. and 9.472 per cent. respectively. The solutions contained both silica and alumina. With sodic carbonate solution albite lost 1.785 per cent.

In the case of wollastonite the sodium hydrate acts as a solvent of the whole mineral.

On Pentathionic Acid. (*Part II.*) By Watson Smith and T. Takamatke. Vol. XL, p. 162.

This paper opens with a defence of the criticisms of V. Lewes' Spring and Th. Curtius upon the authors' previous paper upon this topic. The authors give a number of details of experiments made to establish their position. They have proved "that the attempt to neutralize pentathionic acid with alkaline earth carbonates simply results in the formation of tetrathionates, with separation of sulphur." They further "conclude that on partial saturation of a pentathionic acid solution with an alkali, say potash, we may get a certain amount of potassium pentathionate formed, together with the realization of Stingl and Morawski's equation, $5\ H_2S_5O_6 + 10\ KOH = 5\ K_2S_4O_6 + 5\ S + 10\ H_2O$.

On Some Constituents of Resin Spirit. By G. Harris Morris, F.C.S. Vol. XL, p. 167.

The product obtained from the lower fractions of resin spirit after standing some time with water in large flasks is the subject of this paper. The water solution yields upon evaporation colorless crystals of the composition $C_7H_{14}O_2H_2O$, and with a vapor density 63.6. The crystals are readily soluble in alcohol, ether, benzole. Heated they begin to sublime at 100° C., giving off water. The dehydrated body melts at 89.5 and boils at 195.6° C. without decomposition. The water solution is neutral to test paper, and is not precipitated by basic lead acetate and ammonio-silver nitrate. The crystals warmed with hydrochloric acid and added to alcohol give a fine rose color, which is very characteristic.

The crystals are obtained in the largest quantity from the fraction boiling between 100—105° C., although the fractions from 94—150° C. yield crystals.

A hydrocarbon from resin spirit boiling at 103—104° C. was examined and found to be a heptine with a vapor density of 94.23. By the action of nitric acid a dinitro-heptylene was obtained having the formula $C_7H_{12}(NO_2)_2$; it is easily soluble in alcohol, ether, and benzole, and insoluble in water. It melts at 182° C. The other products of the action of nitric acid upon heptine are carbonic dioxide, formic, acetic, butyric, and succinic acids.

With potassic dichromate heptine gives carbonic, dioxide, and acetic acids. With potassic permanganate it gives an acid not yet examined, and no carbonate.

The crystals mentioned above give with nitric acid the same products as the hydrocarbon, except formic acid. The dehydrated crystals are considered to be methylpropylallyleneglycol. These gave the diacetate $C_7H_{12}(C_2H_3O_2)_2$, which crystallizes in square plates, melting at 68.5° C., and very soluble in alcohol, ether, and benzole.

A bromine compound $C_7H_{12}Br_4$ was also formed from an aqueous solution of the crystals; and by passing gaseous hydriodic acid into the hydrocarbon, C_7H_3I or moniodide was formed.

On the Preparation of Diethylnaphthylamine. By Bernard E. Smith. Vol. XL., p. 180.

This body is best prepared by heating 10 parts of naphthylamime and 15 parts of ethylbromide, with a small quantity of ethyl alcohol to dissolve the naphthylamine, in a sealed tube for 8 hours, at 120°

C. After purification, diethylnaphthylamine is a pale straw-colored oil, boiling at 290° C., and darkening on exposure to light and air. Analysis gave carbon 84.18 and 84.25, hydrogen 8.78 and 8.97, nitrogen 7.25 and 7.33 per cent.

Diethylnaphthylamine hydrochloride is formed by adding hydrochloric acid to the base. The double platinum salt has the formula $(C_{10}H_7\ (C_2H_5)_2\ HCl)_2\ Pt\ Cl_2$. Nitroso-diethylnaphthylamine is formed by adding sodic nitrite to a glacial acetic acid solution of the base. It crystallizes in reddish-golden scales, soluble in ether, alcohol, and benzine, and melting at 165° C.

On the Action of Sulphuric Acid Upon Diethylnapthylamine at High Temperatures. By Bernard E. Smith, Vol. XL., p. 182.

Author heated 20 grms of the base with 20 grms of sulphuric acid for eight hours, at 190–210°C. After adding excess of barium hydrate, and decomposing the barium salt with hydrochloric acid, the solution was mixed with excess of ammonia, which gave a white flocculent preciptate. This precipitate, crystallized from alcohol, gave colorless needles, blackened by light and air. This body melts at 190°C, and distills at above 360°C without change. Analysis gives $(C_2H_5)_2\ N.C_{10}H_6.C_{10}H_6N\ (C_2H_5)_2$ as the formula. It is very soluble in hot alcohol, not very soluble in ether, but readily so in benzine and chloroform. The hydrochloride is readily formed from the base.

The solution from the barium precipitate above mentioned, gave an acid with the formula, $HOO_2SC_{10}H_6N\ (C_2H_5)_2SO_2OH$.

On the Action of Carbon Oxydichloride (*Phosgene gas*) **upon Diethylnaphthylamine.** By Bernard E. Smith. Vol. XL., p. 185.

The base was dissolved in dry benzine, and the solution saturated with phosghene gas ; a white crystalline mass separated out, which, after evaporating the benzine, was treated with hot water, and dilute hydrochloric acid. The latter only dissolved out the hydrochloride of the original base. The insoluble residue was dissolved in alcohol and crystallized. Fractional distillation gave three distinct bodies: 1. Colorless, transparent body, crystallizing in quadratic prisms, melting at 70° C; with the formula $COCl.C_6H_4\ N(C_2H_5)_2$. 2. A colorless, crystalline body, crystallizing in hexagonal prisms, melting at 225°C, and isomeric with the first body. 3. A colorless

crystalline body, crystallizing in rhombs with high refracting power, and melting at 130°C, and containing no chlorine.

By the action of the bodies containing chlorine upon the original base, a compound like the third body $C_{44}H_{47}N_3O_2$ was obtained.

Contributions to the Chemical History of the Aromatic Derivations of Methane. By RAPHAEL MELDOLA. Vol. XL., p. 187.

Author gives the results of the action of benzyl chloride upon diphenylamine. He concludes that the crude products of the action are derivations of triphenylmethane and diphenylmethane. An account of the preparation of diphenylamine green on a large scale is given, and the formation of an alkaline salt of its sulphonic acid. The crude product was called "Viridine," and the salt of the sulphonic acid an "alkaline green. The constitutional formula of the hydrochloride is given, and the empirical $C_{31}H_{25}N_2Cl$. The formula of the base was found to be $C_{31}H_{26}N_2O$. Author mentions other methods of obtaining the green coloring matter; and compares its absorption spectrum with the absorption spectra of methyl and malachite green. In speaking of the sulphonic acids of diphenylamine, the author mentions two of them, a mono- and a di-sulphonic acid. The chloride separates from the alcoholic solution, containing hydrochloric acid, in a granular state; and the base separates from the alcoholic solution, on adding ammonia, as a gummy mass which becomes solid.

Of the derivations of diphenylmethane formed simultaneously with diphenylamine green, the author mentions a white amorphous powder, melting about 89°C., and giving no color upon oxidation. This proved to be phenylamidodiphenylmethane $C_{19}H_{17}N$. It is soluble in benzine, chloroform, ether and carbon disulphide; insoluble in glacial acetic acid, alcohol and acetone.

By heating diphenylamine with two molecules of benzyl chloride and zinc chloride, the author obtained benzyl-phenylamidodiphenylmethane $C_{26}H_{23}N$.

By the action of benzyl chloride upon aniline, the author dibenzylamido-diphenyl-methane $C_{27}H_{25}N$, which resembles the above-mentioned bodies in appearance, is less soluble in benzine and tolerene, and its solutions give blue fluorescence.

The preceding reaction repeated with acetanilide shewed replacement of the acetyl group by benzyl. All the diphenylmethane derivations in this paper are white amorphous powders. The paper is

full of the most interesting theoretical considerations and constitutional formulæ.

Contributions to the Chemistry of Cerium Compounds. By W. N. HARTLEY, F. R. S. E. No. XL., p. 202.

Author prepared pure cerium salts by two methods, that of Mosander and that of Bunsen and Bahr. The purity of the salts was determined by comparing photographs of spectra of 10 per cent. solutions of cerium, didymium, and lanthanum, on the same plate.

A very delicate test for cerium is given thus: To the solution to be examined, either neutral or slightly acid, add ammonia acetate and a little hydrogec peroxide. Much cerium if present gives a brown color, and shaking precipitates gelatinous ceric acetate. With a small quantity of cerium the liquid turns yellow without a precipitate forming. By this method one part of cerium can be separated from 100,000 parts of liquid.

The preparation of cerous phosphate is best performed by adding sulphurous acid to the cerous solution, and then adding an acidified solution of sodium phosphate; gently warming causes the precipitate to settle rapidly. The precipitate is white, and dries to a porcelain-like mass, very hard to pulverize. Author makes the composition $CePO_4$, 2 H_2O.

Ceric phosphate was prepared from solutions of ceric nitrate and sulphate, and although the conditions were varied the composition was the same, and the author concludes that there is but one ceric ortho-phosphate $(Ce\ O_2)_4\ (P_2O_5)_3$.

Tne Analysis of Rhabdophane, a New British Mineral. By W. H. HARTLEY, F. R. S. E. No. XL., p. 210.

The mineral has the general composition $R_2O_3 . P_2O_5 . 2\ H_2O$, in which Ce, Di, La, or Yt may wholly or in part replace each other. Author's analysis gave water (loss on ignition) 9.34—silica 0.36—Al_2O_3, Fe_2O_3, with P_2O_5 0.21—magnesium phosphate 1.09—cerous oxide 23.19—lanthanum and didymium oxides 34.77—yttrium oxide 2.09—phosphoric anhydride 24.77 per cent. Author gives the results of a most careful search for yttrium, with tables of the lines obtained in the spectrum, using a Rutherford's grating with 17,460 lines to the inch. There was a complete absence of lines attributable to erbium; but there were four lines in green which the author could not connect with any known element.

This is the first instance on record of yttrium being found in any British mineral.

On Benzyl-Phenol and its Derivations. (*Part II.*) By Edward H. Rennie, M.A., B.Sc. No. XL., p. 220.

Author describes the following bodies: *Benzyl-phenol—sulphonic acid;* not obtained in a pure state as it loses weight at 100° C and partially decomposes. *Mono-nitro-benzyl-phenol,* golden yellow prisms, melting at 74-75 C°, volatile in steam. The potassium derivative, in needles, have a brick-red color. *Amido-benzyl-phenol;* the hydrochloride forms glistening scales, which are unstable. *Dinitro-benzyl-phenol;* crystallized from alcohol; it melts at 87–88° C. The potassium derivative crystallizes in orange needles and yields benzoic acid with chromic liquor. *Para-nitro-benzoic-acid* was obtained by oxidizing trinitro-benzyl-phenol with chromic liquor; it melts at 238° C. *Nitro-bromo-benzyl-phenol* prepared from potassium bromo-benzyl-phenol-sulphonate and crystallized from alcohol, gives brilliant yellow scales, melting at 64–65° C. The potassium derivative crystallizes in brilliant red scales. The above sulphonate may also be prepared from nitro-benzyl-phenol-sulphonate by treating with bromine in a glacial acetic acid solution. Several other ways of preparing this body are also given. By acting on the above-mentioned nitro-bromo compound with nitric acid, *Dinitrobromphenol* was obtained, which melts at 118° C., and crystallizes from alcohol in fine, pale yellow needles.

In discussing the constitution of benzyl-phenol and its derivations the author gives the results of experiments upon the oxidization of benzyl-phenol-methyl ether. From this ether he obtained *Methoxy-benzophenone* in four-sided prisms, melting at 61–62° C. From paraoxybenzophenone and methyl iodide he obtain the same body, and hence concludes that it is paramethoxybenzophenone, and that it proves that benzyl-phenol is a para-derivative.

Abstracts from *Berichte der Deutschen Chemischen Gesellschaft,* by L. H. Friedburg.

Bromo-substitution Products of Sebacylic Acid. Fr. Gantter and Carl Hell. (Vol. XV., p. 142.) It seems, according to these researches, that red phosphorus acts "catalytically." between bromine and sebacylic acid.

Synthesis of the Homologous Phenols. AD. LIEBMANN (Vol. XV., p. 150.) Preparation of isobutyl-phenol, amyl-phenol, butyl-phenol, and benzyl-phenol.

Determination of Sulphurous Acid in Wine. B. HAAS. (Vol. XV., p. 154.)

Lecture Experiments. MAX. ROSENFELD. (Vol. XV., p. 160.) Use of glass tubes for showing the decomposition of water by red-hot iron.

Decomposition of steam by magnesium.

Combustion of ammonia in oxygen.

On the Etherification of the Oxyacids. N. MENSCHUTKIN. (Vol. XV., p. 162.)

A Synthesis of Thymol out of Cuminol. OSCAR WIDMANN. (Vol. XV., p. 166.) Cuminol is transformed into nitro-cuminol, the latter treated with phosphorus pentachloride, is changed into nitro-cymylen-chloride; zinc, and sulphuric acid as reducing agents, hydrogenizing the nitro-chloro compound into cymidin. Nitrite of potash then transforms the cymidin into nitrosothymol, which was proved to be identical with nitrosothymol, prepared from thymol and nitrous acid in the same manner as previously indicated.

This is the first synthesis of a derivative of thymol, hence of thymol.

Preparation of Succinic Acid from Tartaric Acid by Fermentation. F. KONIG. (Vol. XV., p. 172.) The fermenting liquid is a solution of tartrate of ammonia. Acetic and formic acids are formed in varying quantities, also carbonate of ammonia. The process is recommended as a good one for making succinic acid, even on a large scale.

Preparation of Coloring Matter by the Reaction of Aromatic Nitro-substances on Phenols and on Poly-atomic Alcohols in Presence of De-hydrating Agents. HEINRICH. BRUNNER. (Vol. XV., p. 174.) A representative of the first-mentioned series is prepared by reaction between resorcin and nitrobenzol. Of the second class two combinations have been formed, respectively from erythrit and nitro-alizarine, and cane sugar and nitro-alizarine.

Oxy-propyl-toluidin. R. F. MORLEY. (Vol. XV., p. 179.)

Mono-phenyl-boro-chloride and some of its Derivatives. A. MICHAELIS and P. BECKER. (Vol. XV., p. 180.) The mono-phenyl-boric acid has only a mild toxic influence upon the human system, while it is comparatively poisonous for lower organisms, bacteria, &c. The free acid is about ten times stronger as an antiseptic than its sodium salt.

OnTolyl-Methyl-Keton. A. MICHAELIS. (Vol. XV., p. 185).

Reaction between the additional products of Chinolin plus halogen-alkalies and oxide of silver. W. LACOSTE. (Vol. XV., p. 186).

Isolation of basic compounds, corresponding to a quarternary oxide of ammonium, by means of the above named reaction.

Reaction of Dimethyl-phenyl-phosphine upon Bromide of Ethylene. L. GLEICHMANN, (Vol. XV., p. 198).

Researches on the Laws of substitution in the Naphthalene series. HENRY E. ARMSTRONG. (Vol. XV., p. 200).

Formation of basic compounds from acido-amides. O. WALLACH. (Vol. XV., p. 208).

Influence of heat upon Thio-formanilide. W. W. I. NICHOL. (Vol. XV., p. 211).

Syntheses by means of Phenyl-acetylene and its derivatives. ADOLF BAEYER and LUDWIG LANDSBERG. (Vol., XV., p. 212).

On the question of identity between the coloring matter of the Sophora Japonica Capparis Spinosa and Ruta Graveolens, with Quercitrin and Quercetin. P. FOERSTER. (Vol. XV., p. 214).

Decomposition of mono-halogenized crotonic acids by alkalies. R. FRIEDRICH. (Vol. XV., p. 218).

Analyses of some Nephrites of Lake Dwellings. K. SEUBERT and G. LINK. (Vol. XV., p. 219).

The proportions of silicic acid to bases and to water lead to the general formula $R^{II}\ Si\ O_3$ of the group of hornblende. They, therefore, belong to the group of veritable nephrit (Cacholong of *Rammelsberg*): occurrence: Maurach on the Bodensee.

On rendering oxygen active. MORITZ TRAUBE. (Vol. XV., p. 222).

Palladium-hydrogen shaken with water and oxygen (air) yields immediately and abundantly peroxide of hydrogen. The oxidizing effects of palladium-hydrogen in presence of oxygen and water are not directly caused by the former, but almost exclusively by the peroxide of hydrogen generated. Potassium iodide starch mixture is excepted. Peroxide of hydrogen does not turn this blue, while palladium-hydrogen and oxygen do. Here palladium transfers the oxygen from peroxide of hydrogen to the iodide of potassium. Nascent hydrogen is not able to render oxygen active by splitting its molecules.

On Anthramin. C. LIEBERMANN and A BOLLERT. (Vol. XV., p. 226).

Anthramin is Liebermann's name for the same compound, which Roemer calls anthracylamin=C_{14} H_9 NH_2, and which has been found simultaneously by both gentlemen, independent of each other, in the same laboratory. Anthramin is prepared from anthrol by amidizing it with acetamid in a closed tube at + 280° C., and subsequent distillation of the products of the reaction with potassium hydrate and water. Yield: Twenty per cent of the anthrol.

Cause of Jorissen's reaction for Fusel Oil. K. FOERSTER. (Vol. XV., p. 230).

Fusel oil, heated with aniline and hydrochloric acid, produces a beautiful red coloration of the mixture. On this fact Jorissen has founded a method for testing common alcohol for fusel oil (produced by fermentation).

Foerster's investigation shows that this coloration is due to an impurity, and that neither amylic alcohol nor its homologues cause the reaction.

The impurity was found to be furfurol, of which the property of producing color-reactions with bases of the aromatic series is well known.

The combinations of Benzo-tri-chloride with aromatic Bases. OSCAR DOEBNER. (Vol. XV., p. 232).—The author published, some time ago, his researches on the action of benzotrichloride and phenols, as well as on tertiary aromatic bases, now he deals with the primary and secondary aromatic bases, beginning with aniline. New dyeing substances are among the results of this investigation

Abstracts from the *Comptes Rendus*, by A. Bourgougnon.

Double Salts formed by the Haloid Salts of Mercury. By M. BERTHELOT. (No. 7, Feb. 13th, 1882.

Researches upon the Nitrogenized Acids derived from the Acetones. By G. CHANCEL. (No. 7, Feb. 13th, 1882).

By the action of nitric acid upon propion and upon butyron, ethylnitrous acid, CH_3,—$C N_2 O_4 H$, and propylnitrous acid, $C_2 H_5$—$CN_2 O_4 H$, are formed. These two compounds are identical with dinitro-ethan and dinitro-propan. The characteristics of all these nitrogenized acids is the univalent group

$$C^1 N_2 O_4 H = C^1 \begin{cases} -NO_2 \\ -NO_2 \\ -H, \end{cases}$$

of which the saturation is completed by one atom of hydrogen (methylnitrous acid) or by an alcoholic radical; the general formula of alkylnitrous acids is then

$$\begin{matrix} C^n H^{n+1} \\ C^1 N_2 O_4 H \end{matrix}$$

By the action of reducing agents the group $C N_2 O_4 H$ is transformed into ammonia, hydronylamin and $C O O H$, characteristics of the organic acids.

The formation of alkylnitrous acids by the action of nitric acid upon the acetones is represented as follows :

$$\underbrace{C_7 H_{14} O}_{\text{Butyron.}} + 2\ NO_3 H = \underbrace{C_3 H_6 N_2 O_4}_{\text{Propylnitrous acid.}} + C_4 H_8 O_2 + H_2$$

Amylnitrous acid, $C_4 H_9$—$CN_2 O_4 H$. Pentyl or amylnitrous acid is obtained by the reaction of nitric acid upon normal capron.

Amylnitrite of Potassium, $C_4 H_9$—$CN_2 O_4 K$; yellow needles of fatty appearance.

Amylnitrate of Silver, $C_4 H_9$—$CN_2 O_4 Ag$ is like the potassium salt, but less soluble.

Butylnitrous acid, $C_3 H_7$—$CN_2 O_4 H$. Obtained by the action of nitric acid upon the most volatile part of crude capron, distilling at 175° C. Uncolored liquid, heavier than water, transformed into normal butyric acid by reduction.

Butylnitrite of Potassium, $C_3 H_7$—$C N_2 O_4 K$. Yellow prisms.

Butylnitrite of Silver, $C_3 H_7$—$C N_2 O_4 Ag$. Crystalizes with difficulty from a warm saturated solution.

Herylnitrous acid, $C_5 H_{11}$—$C N_2 O_4 H$. Obtained by the action of nitric acid upon methylenanthol,

or hexylmethylacetone, $C_6 H_{13}$—$C O$—$C H_3$
potassium salt $C_5 H_{11}$—$C N_2 O_4 K$
silver salt $C_5 H_{11}$—$C N_2 O_4 Ag$.

PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

Room 1, *University Building, N. Y.*

The last regular meeting was held Friday, October 6, 1882. The meeting was called to order at 8.45 P. M., Mr. P. Casamajor in the chair.

The minutes of the previous meeting were read, but as there was not a quorum present, they could not be acted upon.

After which, the first paper of the evening, "On the fractional dehydration of Ammonium Alum and the Atomicity of Aluminium," by Prof. J. W. Mallet, was read.

This paper describes a large number of interesting experiments, which go to show that Aluminium is either a triad or pentad.

After some remarks, the second paper "On the determination of Sulphur in Gas," by Mr. A. P. Hallock, was read.

Mr. Hallock stated that he used dehydrated sulphate of copper for the estimation of H_2S.

The apparatus used, consisted in a series of U tubes; the gas being allowed to pass through them for a certain length of time.

Dr. Grothe then inquired whether the above gas contained any Ammonia? Mr. Hallock stated that it did, but only in traces.

Dr. Grothe further asked whether the above gas contained any acetylene? Mr. Hallock replied that there was none. Upon which Mr. Casamajor remarked that possibly sheet rubber would remove small traces of hydrocarbons.

Mr. Elliott then stated that the Municipal Gas contains no other hydrocarbons than CH_4, and that this is not absorbed by the dehydrated sulphate of copper.

He further stated that all the sulphur in water gas is in the shape of sulphuretted hydrogen.

Some remarks about the combustion of sulphur in gas, lead to a lively discussion, in which Mr. Elliott stated that the H_2S was all burnt to H_2SO_4, while Dr. Grothe and Mr. Casamajor insisted that SO_2 is likewise formed.

The following gentlemen were then nominated:

Prof. Cuthbert P. Conrad, regular member. Proposed by J. W. Mallet, E. Waller and James H. Stebbins, Jr.

Ervin von Wilmouski, regular member. Proposed by James H. Stebbins, Jr., Wm. Rupp and Dr. Grothe.

Alfred L. Beebe, regular member. Proposed by J B. McIntosh, A. L. Colby and A. H. Elliott.

John Cawley, regular member. Proposed by James H. Stebbins, Jr., A. H. Elliott and P. Casamajor.

N. Hathaway, regular member. Proposed by E. Waller, Wm. Rupp and C. E. Munsell.

Geo. Leguin, associate member. Proposed by Nelson H. Darton, James H. Stebbins, Jr., and Wm. Rupp.

John G. Macfarlan, associate member. Proposed by P. Casamajor, James H. Stebbins, Jr., and A. H. Elliott.

Dr. Waller then stated on behalf of the committee on papers, that the lack of MS. produced much trouble, and he invited the members to assist said committee, by supplying as much MS. or abstracts as possible.

There being no further business the meeting adjourned.

JAMES H. STEBBINS, JR.,

Recording Secretary.

Laboratory Notes.—By E. Waller.

PRECIPITATION OF BARIUM SULPHATE.

I have frequently observed that solutions of barium chloride seem to dissolve more air or carbon dioxide than most other solutions used as reagents, so much so that the addition of a cold barium solution to a boiling solution causes a brisk evolution of gas comparable with the effervescence due to the addition of acid to a carbonate. When a sulphate is present in the solution to which cold barium chloride solution has been added, even though the sulphate solution may have been boiling, the precipitate separates in a very finely divided condition, which gives much trouble in filtering, as the precipitate is so fine as to pass through the pores of a filter, and takes very long to settle.

If the barium solution is boiled for a few minutes before adding it, this phenomenon does not occur. The conclusion seems to be that the gases dissolved by the barium solution play an important part in the matter of the separation of the precipitate. Other chemists have evidently experienced the same trouble, for I have found recommendations in the literature to add a pinch of starch to the solution, and also more recently the addition of a little silver

nitrate, the Ag Cl. being subsequently dissolved out by ammonia, both having the same object in view,—to cause the barium sulphate to separate promptly in a form suitable for filtration and washing.

However, by boiling *both* barium and sulphate solutions for a few minutes *separately* and mixing them when boiling hot, then boiling the mixture for a few minutes and finally allowing it to stand on the water-bath, the precipitate will settle completely in about half an hour.

PECULIAR REACTION WITH LEAD ACETATE.

Many of the members of the Society will remember a conversazione of the Society which was held at the laboratory of our late fellow member, Dr. Lipps. Among other interesting things shown us was the preparation of Dr. Lipps' "dextrin reagent," where litharge was added to solution of lead acetate, and the mixture heated to about 55°C., when it thickened up, owing, no doubt, to the formation of some of the extremely basic lead compounds. The reaction is such a peculiar one that I have thought it worthy to be placed on record, which Dr. Lipps failed to do during his lifetime.

To obtain the reaction, take a cold saturated solution of lead acetate, warm to 50 or 60°C., and add litharge little by little, stirring it in vigorously. At a certain point the mixture suddenly thickens, and in a few minutes becomes so solid that the vessel may be inverted without spilling. After standing for a short time the mass may be extracted with water, and the solution constitutes the "dextrin reagent." This reagent, when boiled with solutions containing dextrin, gives a white precipitate. According to Dr. Lipps 100 parts of this precipitate, when thoroughly dried, corresponds to 35 parts of dextrin.

The compound formed is probably the sexbasic lead acetate described by Berzelius (Ann. Chim., XCIV., 296), containing—

Acetic acid	5.7 per cent.
Oxide of lead	91.3 "
Water	3.0 "

In Berzelius' description of the preparation of this acetate, ammonia was used to withdraw a portion of the acid from its combination with the lead. In Allen's Commercial Handbook, p. 318, the use of lead acetate to which ammonia has been added is mentioned as a reagent for dextrin, The compound formed is probably the same.

Laboratory Notes.—By JAMES H. STEBBINS, JR., S. B., F. C. S.

ANALYSIS OF GALENA FROM NEWBURYPORT, MASS.:

This ore was said to be rich in silver; however, upon analysis, only a trace could be found, while the percentage of PbS is quite large.

The composition of this ore may be seen from the following figures:

SiO_2	0.40
PbS	98.80
Sb_2S_3	0.40
FeS	0.15
Ag. trace	....
Total	99.75

Its appearances closely resembled ordinary galena, having a cubic cleavage and coarse structure. Hardness about 2.5; lustre, metallic; and streak, lead gray.

ANALYSIS OF PYRRHOTITE FROM CLAYTON, N. Y.:

This ore was handed to me to be tested for gold, but, upon analysis, no gold was revealed.

Its appearance was massive and amorphous. Hardness, about 3.5—4; lustre, metallic; color, between bronze-yellow and copper-red. Several of its sides were tarnished. Streak, dark grayish black. It was, moreover, strongly magnetic when reduced to a fine powder.

Composition.

SiO_2	3.30
Fe	58.03
S	38.26
P_2O_5 trace	....
Total	99.59

According to theory, it should have the following composition:

Calculated for Fe_2S_3.

Sulphur	39.5
Iron	60.5
Total	100.0

It will, therefore, be observed that the above ore deviates but slightly from theory.

ANALYSES OF THE CALCULI IN THE "WOOD" MUSEUM OF BELLEVUE HOSPITAL.

By A. Bourgougon.

In 1867 the late Dr. Jas. R. Wood deeded to the City of New York a most complete and valuable collection of anatomical specimens. Among other curiosities there were many calculi. Some of these deserve particular attention, from the fact that they have been removed by Dr. Wood; others because of their size or composition.

A classification of the specimens could not be made unless analysis had determined their composition, and with this view they were submitted to chemical tests.

As chemists, especially those interested in physiological chemistry, may not be aware of the existence of this Museum, the results of the analyses are submitted in a condensed form. The numbers attached to each specimen indicate those of the catalogue of the Museum.

970. Stone removed by Dr. J. R. Wood at his clinic at Bellevue Hospital, Jan. 10th, 1880. Several pieces, 13 grammes. A nucleus of wax and dried blood surrounded by layers of phosphate of ammonium and magnesium.

971. Weight, 70 grammes. Calcium oxalate, urate and phosphate.

972. 5 stones; weight, 94 grammes. Ammonio-magnesium phosphate, calcium urate.

973. Weight, 58.5 grammes. A nucleus of uric acid, ammonium urate, calcium urate and silica, surrounded by calcium phosphate and amm. magn. phosphate.

974. Weight, 63 grammes. Calcium urate, oxalate and phosphate.

975. Weight, 48 grammes. Calcium oxalate and urate.

976. Weight, 26.5 grammes. Calcium oxalate and phosphate.

977. Weight, 49.5 grammes. Mulberry calculus, well formed. Calcium oxalate and urate. The nucleus contains ammonium urate.

978. Weight, 34.5 grammes. Removed from a colored man at Bellevue Hospital. Uric acid, amm. magnesium phosphate, ammonium urate, calcium oxalate.

979. Weight 9 grms. A nucleus formed by a candle or lamp wick. Surrounded by uric acid, calcium urate and phosphate.
980. Weight 15 grms. Uric acid, calcium oxalate and urate.
981. Weight 11.5 grms. Mulberry calculus, calcium oxalate.
982. Weight 15 grms. Cholesterin (biliary.)
983. Weight 15 grms. Calcium oxalate, calcium phosphate.
984. Weight 22 grms. The patient was over 70 years of age. Amm. magnesium phosphate.
985. Weight 25.5 grms. Removed by Dr. J. R. Wood. Calcium oxalate and urate.
986. Weight 26 grms. Removed from a boy 13 years old, by Dr. J. R. Wood, Saturday, Nov. 3d, 1877, at Bellevue Hospital. Uric acid, calcium urate and oxalate.
987. Weight 29.5 grms. Only one-half of the calculus. Uric acid, calcium urate, and phosphate and silica.
988. Weight 21 grms. Removed Dec. 13th, 1877. The patient was 70 years old. Calcium phosphate.
989. Weight 36 grms. A nucleus of ammonium urate, surrounded by calcium phosphate, amm. magnesium phosphate.
990. Weight 8 grms. 6 stones removed from a boy 6 years old. Calcium phosphate and urate.
991. Weight 12 grms. Calcium oxalate.
992. Weight 6 grms. 7 stones removed from a boy 12 years old. Calcium phosphate and amm. magn. phosphate.
993. Several small pieces. Patient 22 years old. Calcium urate and oxalate.
994. Calcium urate and oxalate.
995. Uric acid, amm. magn. phosphate.
996. Weight 25 grms. Dried blood and calcium phosphate.
997. Weight 20 grms. Removed Nov. 15th, 1868. Patient 20 years old. Uric acid, calcium urate, amm. magn. phosphate.
998. Weight 20 grms. Removed Dec. 16, 1872. Uric acid, amm. magn. phosphate.
999. Weight 32 grms. Removed May 14, 1874. The patient was 64 years old. Amm. magn. phosphate.
1000. Weight 31 grms. Calcium urate and phosphate, and amm. magn. phosphate.
1001. Calcium oxalate and urate.

1002. Weight 20 grms. Patient 40 years old. Calcium phosphate and oxalate.
1003. Amm. magn. phosphate.
1004. Uric acid, amm. magn. phosphate, and calcium phosphate.
1005. Uric acid and amm. magn. phosphate.
1006. Uric acid, calcium urate and phosphate and silica.
1007. Amm. magn. phosphate calcium oxalate and phosphate.
1008. Amm. magn. phosphate and calcium phosphate.
1009. Calcium oxalate and phosphate.
1010. This is not a calculus, it is a water-worn stone, a kind of silex, which ought to be removed from the collection.
1011. Amm. magn. phosphate and calcium phosphate.
1012. Uric acid and calcium oxalate.
1013. Uric acid.
1014. Amm. magn phosphate, calcium urate, and uric acid.
1015. Uric acid, amm. magn. phosphate.
1016. Calcium urate, amm. magn. phosphate.
1017. Calcium urate and phosphate.
1018. Uric acid, calcium oxalate, amm. magn. phosphate.
1019. Calcium oxalate.
1020. Calcium oxalate and phosphate, and amm. and magn. prosphate.
1021. Calcium urate, phosphate, and amm., magn. phosphate.
1022. Calcium urate and amm. magn. phosphate.
1023. Two calculi, amm. magn. phosphate and calcium phosphate.
1024. Removed Dec. 13, 1870, at Bellevue Hospital. Calcium oxalate, amm. magn. phosphate.
1025. Removed from a child 2 years and 9 months old, by Dr. J. Wood. Uric acid and amm. magn. phosphate.
1026. Uric acid, calcium urate and silica.
1027. Uric acid, calcium phosphate.
1028. Calcium urate, amm. magn. phosphate.
1029. Amm. magn. phosphate, calcium phosphate and calcium urate.
1030. Calcium urate, amm. magn. phosphate.
1031. Uric acid.
1032. Amm. magn. phosphate, calcium oxalate.
1033. Amm. magn. phosphate.
1034. Removed Nov. 2d, 1872, by Dr. J. R. Wood, from a child 6 years old, at Bellevue Hospital. Calcium oxalate.
1035. Calcium oxalate.
1036. Two calcali, amm. magn. phosphate.
1037. Calcium oxalate, amm. magn. phosphate, calcium phosphate.

1038. Several small pieces removed from the bladder by lithotrity. Uric acid, ammonium urate, amm. magn. phosphate.

1039. Several small pieces uric acid, amm. magn. phosphate.

1040. Uric acid.

1041. Cholesterin.

1042. Several pieces weighing 18 grms., removed by Dr. Ch. Phelps, May 9th, 1877, from a man 66 years old. Amm. magn. phosphate, calcium phosphate.

1043. Nucleus of a calculus removed by Dr. Ch. Phelps Nov. 30th, 1877. A pin surrounded by dried blood, uric acid and calcium phosphate.

1044. Several pieces calcium oxalate and phosphate.

1045. Renal calculus. Uric acid, amm. magn. phosphate.

1046. Renal calculus. Amm. magn. phosphates. Among the pieces of this calculus are three stones of cholesterin; they are here evidently by mistake.

1052. Debris from operation by lithotrity; patient 40 years old. Amm. magn. phosphate, calcium phosphate.

1056. Vesical calculus of horse, only one-half, weight 180 grms. Calcium carbonate (nucleus), amm, magn. phosphate.

Intestinal calculus of horse, of a spherical form, weighing 390 grms. (no number in the collection) has the following composition:

Organic matter.....	6.13	per cent.
Silica	0.24	"
Amm. magn. phosphate.....	86.24	"
Calcium carbonate.....	2.03	"
Magnesium.....	5.25	"
	100.00	

Before the analysis was made the powder of the calculus was dried at 100°C.

1057. Weight 25 gms. Cholesterin. Removed from (?) vagina of a woman 30 years old.

ABSTRACTS.

Abstracts from the *Journal of the Chemical Society*, London, by Arthur H. Elliott, Ph. B., F. C. S.

On the Solubility of Glass in Certain Reagents. By Richard Cowper, A. R. S. M. (Vol. XL., p. 254.)

Author kept a measured quantity of each reagent in a sealed tube of hard glass for six days at 100°C. Two series of experiments, A. and B., gave the results shown in table below, on neutralizing with hydrochloric acid and evaporating to dryness and igniting in a platinum dish.

Reagent.	No. Mgrms. dissolved by 100 cc. A.	B.
Water	8.0	10.0
H_2S	12.5	8.7
$(NH_4)_2S$ dilute, preprared from NH_4OH of sp. gr. 0.982	49.6	52.5
$(NH_4)_2S$ concentrated, prepared from NH_4OH of sp. gr. 0.88	34.	47.2
NH_4OH dilute, sp. gr. 0.982	25.8	42.5
NH_4OH concentrated, sp. gr. 0.88	7.5	7.7
NH_4HS, prepared from NH_4OH sp. gr. 0.982	——	51.2

Another experiment in which dilute $(NH_4)_2S$ was boiled in a flask, 60 cc., gave 0.008 grm. residue.

A series of experiments to find the degree of dilution of Am OH for maximum effect gave following results:

40 cc., taken.	Dissolved in 100 cc.
NH_4OH sp. gr. 0.88+1 vol H_2O	22.5
" " " " +2 vols "	26.2
" " " " +3 " "	30.0
" " " " +4 " "	26.7
" " " " +5 " "	41.2
" " " " +6 " "	32.0

Analysis of a Piece of Oxidized Iron from the Condenser of H. M. S. "Spartan." (Vol. XL., p. 256.) By Richard Cowper, A. R., S. M.

The details of the methods of analysis are given, and the following results:

Insoluble Residue dried at 120° C	30.14	C	12.57	SiO_2	16.98
		H	.24	Fe_2O_3	.12
		Mineral	17.54	Al_2O_3	.06
				CaO	.15
				MgO	.02

Cupric oxide	.38
Ferric "	2.21
Ferrous oxide	42.33
Alumina	.16
Manganic oxide	1.02
Cobalt oxide	.05
Sodium oxide	.11
Phosphoric acid	5.24
Sulphuric "	.31
Vanadic "	.11
Chlorine	2.08
Water	16.71

Note on a Convenient Apparatus for the Liquefaction of Ammonia. (Vol. XL., p. 259.) By J. Emerson Reynolds, M. D., F. R. S.

The apparatus consists of two vertical wrought iron tubes joined below by a horizontal tube of the same metal. The vertical tubes are 40 and 30 cm. high and 12 cm. internal diameter. The horizontal tube is 25 cm. long and 5 or 6 cm. internal diameter. The higher vertical tube has a gas-tight screw cap. The lower vertical tube also has a tight-fitting cap, which is perforated to receive the slender stem of a tube very much like a thermometer, which is cemented into this cap. The tube in the cap is about 25 c. m. long and the bulb below terminates in a beak which is always open. To fill the tube with dry ammonia gas, both ends are left open, and the gas allowed to flow through to expel all air. Now the upper end is sealed by a blow-pipe, and the lower beak dipped into mercury, which enters and prevents the escape of the gas.

The cap of the highest vertical iron tube is now removed and the iron apparatus mentioned above is filled with mercury until the lower tube is nearly full. The glass tube with the ammonia gas is now placed in the lower iron tube, and the cap which holds the glass tube is screwed tightly home, with a leather washer. From the higher

iron tube remove enough mercury to leave about 12 cm. below the surface of the cap ; fill this space with strongest liquor ammonia and screw the cap into its place. By heating carefully the higher iron tube containing the ammonia solution, a pressure is exerted upon the glass apparatus in the lower tube and the ammonia gas is liquified. This apparatus may be used for other gases.

Transformation of Urea into Cyanamide. (Vol. XL. p. 262) By H. J. H. Fenton, M.A.

Author dehydrates urea by heating with metallic sodium and obtain cyanamide according to the following reaction. $2\ CON_2H_4+Na_2=2CN_2H_2+2NaOH+H_2$. Ammonium carbamate and ammonium carbonate behave similarly under like circumstances.

On the action of Haloid Acids upon Hydrocyanic Acid. (Vol. XL. p. 264). By L. Claisen, Ph. D. and F. E. Matthews, F.C.S.

After giving details of their experiments, the authors, conclude that, "when hydrocyanic acid unites with haloid acids in presence of ethers, a simple addition compound is not formed, but a more complex one, 2 mols. of hydrocyanic acid uniting with the haloid acid." The body $2HCN+3HCl$. when acted upon by alcohol gave *formamidine.*

On the action of Acetyl Chloride on Fumaric Acid. (Vol. XL. p. 268). By W. H. Perkin, F.R.S.

This paper gives some results of experiments in answer to the criticisms of Anschütz (Ber. 14, 2792) and C. Bennert (Ber. 15, 640). The author concludes that acetyl chloride acts upon fumaric acid in the same way as it does on other dibasic acids, forming the anhydride by the removal of a molecule of water, the anhydride in this case being maleic anhydride ; and as a secondary result, part of the maleic anhydride thus formed unites with the hydrochloric acid present and forms chlorosuccinic anhydride.

On the Action of Acetone on Phenanthraquinone, both alone and in Presence of Ammonia. (Vol. XL., p. 270.) By Francis R. Japp, M.A., Ph. D., and Fred. W. Streatfield.

Fifty grms. of phenanthraquinone were mixed with 60 grms. of acetone, and 40 cc. of strong aqueous ammonia. The quinone is converted into a white powder, and the temperature rises. When cool, ether was added, and the white powder was washed with ether.

The crude product was dissolved in boiling acetone (b. p. 56-58) containing a little aqueous ammonia. The filtered solution gave colorless rhomboidal laminæ. The fusing point could not be determined accurately; the body changes color at about 120° and fuses about 130°. Analysis leads to the formula $C_{17}H_{15}NO_2$. It dissolves readily in cold concentrated hydrochloric acid, the solution deposits a blue substance which was filtered out, and the filtrate treated with water. Water caused the deposition of fine white needles, which crystallized from ether gave large thin colorless blades, fusing at 89.5-9°, and containing no nitrogen or chlorine. Analysis gave formula $C_{17}H_{14}O_3$, according to the reaction $C_{17}H_{15}NO_2+OH_2=C_{17}H_{14}O_3+NH_3$ since the hydrochloric acid filtrate from needles contains ammonic chloride. The body $C_{17}H_{14}O_3$ is insoluble in water; ether, alcohol and acetone dissolve it freely. In the alcoholic solution boiling converts it into phenanthraquinone; below 40° it can be crystallized from alcohol. By passing NH_3 into the ether solution of the body $C_{17}H_{14}O_3$, the former body $C_{17}H_{15}NO_2$ is obtained according to the reaction $C_{17}H_{14}O_3+NH_3=C_{17}H_{15}NO_2+OH_2$. By heating the body $C_{17}H_{14}O_3$, it is decomposed into acetone and phenanthraquinone. This same body can be formed synthetically by heating phenanthraquinone with acetone in sealed tubes for four hours at 200°.

The authors give a number of theoretical considerations as to the constitution of the body formed, but seem to arrive at no definite conclusions. For the body $C_{17}H_{14}O_3$ they suggest the name *acetonquin of phenanthrene*, and for $C_{17}H_{15}NO_2$ the name *acetonquinimide of phenanthrene*. They will continue their studies on these bodies.

A Study of Some of the Earth-metals contained in Samarskite. (Vol. XL., p. 277.) By Henry E. Roscoe, V.P.R.S.

Author gives the details of experiments upon the separation of terbia, yttria, and the so-called phillippia in the form of formates by fractional precipitation. A very careful series of measurements of of the so-called phillippian formate crystals as rhombic prisms is given, for comparison. A solution of the earths terbia, phillippia and yttria was passed through the processes of fractionation, and no oxide having an unalterable atomic weight of 122 could be detected. Author gives details of experiments which serve to prove that the formates of terbium and yttrium crystallize together in a form ascribed to phillippium.

The Spectrum of Terbium. (Vol. XL., p. 283.) By H. E. Roscoe and A. Schuster.

The spectrum was obtained with "a grating of Mr. Rutherford containing 17,290 lines to the inch, and all measurements were taken in the spectrum of the second order. The focal length of the observing telescope was 17.4 inches, and the measurements were taken by means of a micrometer eye-piece, the screw of which had 100 turns to the inch." A table of the lines observed is given, showing their wave-lengths and relative intensity.

On the Behavior of Zinc, Magnesium, and Iron as Reducing Agents with Acidulated Solutions of Ferric Salts. (Vol. XL., p. 287). By T. E. Thorpe, F.R.S.

In the case of zinc the author states: "It results from these observations that, provided there is sufficient acid present to dissolve the zinc, the maximum reducing action possible with a given weight of metal is obtained by concentrating the ferric sulphate solution and diminishing the amount of free acid. The effect of concentration is to multiply the chances of contact of the hydrogen and ferric sulphate molecules; increase of temperature brings about the same result, by augmenting the molecular movement within the liquid." By using platinum in contact with the zinc, the time of solution is diminished, but very little difference in the reducing effect is observed.

With magnesium the amount of reduction is only $\frac{1}{4}$ to $\frac{1}{3}$ of that effected by zinc under corresponding conditions. The presence of platinum but slightly augments the reducing power of magnesium.

With iron the rate of solution is extremely slow, and the reducing action *apparently* decreases with the temperature. In contact with platinum both the rate of solution and the reducing action are increased; the action is *decreased*, however, on warming the liquid.

The author remarks: "These experiments seem to me to be in entire harmony with the view that the reducing action of so-called nascent hydrogen is connected with the existence of this body in the atomic state, and that any conditions tending to prolong the duration of this atomic condition augment the reducing power of the hydrogen."

Note on the Action of the Oxychlorides of Sulphur on Silver Nitrate. (Vol. XL., p. 297). By T. E. Thorpe, F.R.S.

Thionyl chloride $SOCl_2$ with silver nitrate gives nitrosulphonic chloride and silver chloride. Sulphuryl chloride SO_2Cl_2 has no

action. Chlorosulphonic acid SO_2OHCl acts violently on silver nitrate, forming silver chloride and nitrosulphonic acid (chamber crystals.)

On the Action of Thiophosphoryl Chloride upon Silver Nitrate. (Vol. XL., p. 297). By T. E. Thorpe, Ph. D., F. R. S., and Septimus Dyson, Esq.

By adding thiophosphoryl chloride drop by drop to dry silver nitrate and condensing the products of the action, the authors obtained nitrogen peroxide, nitric oxide and nitroxyl chloride, and *nitrosulphonic anhydride.* The authors say: "We have no hope, therefore, of being able to form a mixed anhydride, on the type of nitric anhydride in which more or less of the oxygen is replaced by sulphur, by the substitution of thiophosphoryl chloride in Odet and Vignon's reaction."

Experiments on the action of Potassium Amalgam Sulphuretted Hydrogen, and Potassic Hydrate respectively, on Tetra and Pentathionate of Potassium. (Vol. XL., p. 300.) By Vivian Lewes, Royal Naval College.

By the action of potassium amalgam upon potassium tetrathionate in solution the thiosulphate ($K_2S_2O_3$) is found when no excess of amalgam is used; in the latter case sulphide is formed. With potassic pentathionate potassium amalgam gives first tetrathionate and sulphur, then thiosulphate.

By the action of sulphuretted hydrogen upon potassic tetrathionate, the thiosulphate is formed and sulphur separates.

By the action of potassic hydrate upon a solution of potassic pentathionate thiosulphate, sulphite and sulphur result from the decomposition. In the case of potassic hydrate and tetrathionate the results are thiosulphate and sulphite.

On the Estimation of Retrograde Phosphates. (Vol. XL., p. 306.) By Fred. J. Lloyd.

Author reviews the work of Fresenius, Neubauer, Luck and others in this field. He then proceeds to find out the relative dissolving powers of citric acid, partly neutralized, wholly neutralized, and more than neutralized by ammonia, upon the phosphates and other substances present in manures. He concludes that no satisfactory determination of retrograde phosphates can be made by an acid or slightly acid solution of ammonium citrate, owing to the powerful action upon other substances. From another set of experiments the author states that an ammonical solution of ammonium citrate, no matter what its

strength, whilst dissolving precipitated calcic phosphate, does not act upon mineral phosphates, bone ash, and but slightly upon raw bones. This paper is a most valuable one, as the author gives a number of details of experiments upon the methods of other chemists, and a comparison with that proposed by himself.

Action of Heat upon Mercuric Chloride under low pressure. (Vol. XL., p. 322). By Thos. Carnelley, D.Sc.

The general results of the author's experiments are: "When mercuric chloride is strongly heated under pressures less that the critical pressure, then the temperatures attained by the salt as indicated by the thermometer, the turpentine calorimeter, and the petroleum or benzine calorimeter, are as follows:

By the thermometer.—As a compact powder the salt rises 21° C., and sometimes 50° C., above the melting point. When the salt is solidified around the thermometer bulb it rises 15° C. above the melting point. *By the turpentine calorimeter.*—the temperature rises 100° C. above the melting point. *By the petroleum calorimeter.*—The temperature cannot be obtained above the melting point; but is generally a few degrees less.

The author concludes that the high temperature registered by the turpentine calorimeter is due to the heat developed by the action of the mercuric chloride upon the hydrocarbon.

On the Constitution of Amarine and Lophine. Vol. XL., p. 323. By Francis K. Japp, M. A, Ph.D. and H. H. Robinson.

From a consideration of Emil Fischer and H. Troshke (Ber. 13. 706) and a comparison with the formula obtained by one of the authors and Mr. Streatland (Jour. Chem. Soc., xl. p. 149) for the compounds formed by the action of hydroxyaldehydes and ammonia upon phenanthraquinone, an unmistakable resemblance was noticed. Acting upon this resemblance, and submitting benzil to the action of parahydroxybenzaldehyde in presence of ammonia, they obtained the body $C_{21}H_{16}N_2O$, which by distillation with zinc dust in a current of hydrogen gave *lophine*. The body $C_{21}H_{16}N_2O$ is *parahydroxylophine*, and lophine is considered to be one of Hubner's anhydro-bases, and is called *anhydrobenzayldiamidostilbene*. From these facts and others connected with amarine, which differs from lophine in that it contains two more atoms of hydrogen, the authors give new constitutional formulæ for both these bodies.

Abstracts from *Berichte der Deutschen Chemischen Gerellschaft*, by Percy Neymann. Ph. B

On a New Combination of Carbon with Sulphur and Bromine. Carl Hell and Fr. Uhech, (Vol. XV, p. 283.)

A mixture of bisulphide of carbon and bromine (the proportion $1CS_2$: $2\ Br_2$ has been found best) after having been left to itself several days and then subjected to a slow distillation leaves a less volatile brownish-red oily residue which does not solidify until brought in contact with water, but then gradually crystallizes. A more rapid method is to cover the oily residue with alcohol instead of with water, or to dissolve the residue in ether and add alcohol. The composition of the new body, termed by the authors carbotrithiohexabromide corresponds to the formula $C_2S_3Br_6$.

The action of concentrated caustic alkali at waterbath temperature and presence of a current of hydrogen to prevent oxidation corresponds exactly to the formula :

$C_2S_3Br_6+12NaOH=2NA_2CO_3+Na_2S_3+6NaBr+6H_2O$. The constitution of the new combination cannot be fixed upon either from the conditions under which it is formed or from the products of decomposition.

On the action of Amines upon Chinones. H. Fincke. (Vol. XV. p. 279), continued from Vol. XIV. p. 1310, derivatives.

On Tropin. G. Merling. (Vol. XV. p. 287), continued from Vol. XIV. p. 1829.

The author starting with 40 grms. tropin obtained only 1 grm. of the neutral oil by the distillation of the dimethyltopinhydroxide. Oxidation of tropin with permanganate of potash in alkaline solution gives rise to a base which by the separation of one atom of carbon as carbonic acid and the formation of a nitroso compound by the action of nitrous acid and also by the regeneration tropin when treated with methyl iodide, must be characterised as a secondary base. Excess of oxidizing agent must be avoided.

Oxidation of tropin by means of bichromate of potash and sulphuric acid or by chromic acid produces (without the separation of carbonic acid) a well characterised dibasic acid of the formula $C_8H_{13}NO_4=(C_6H_{11}N)\begin{matrix}COOH.\\COOH.\end{matrix}$

The author expects at some future day, to be able to contribute something more certain on the constitution of tropin.

On Terebenthenic acid. W. ROSER. (Vol. XV. p. 293).

Sodium reacts with terebenthenic acid ether $C_9H_{14}O_4$ and generates hydrogen; the white crystals which are the product of the reaction are not a sodium combination, analogous to that obtained by J. Bredt* from isocapralacton, but it is the sodium salt of the acid teraconicacidether after liberating the ether by means of acids this yields teraconic acid ($C_7H_{10}O_4$) by saponification which Geissler found among the products of distillation of terebenthenic acid. Terebenthenic acid ether treated in alcoholic solution with one equivalent of sodium ethylate also produces the sodium salt of the acid teraconic acid ether, a second equivalent of ethylate of sodium acts as a saponifying agent upon the primary salt and teraconate of sodium is produced. It was also shown that unless a certain quantity of water was present succinate of sodium was produced. In order to explain the formation of teraconic acid from terebenthenic acid it must be aceepted that by the action of sodium upon terebenthenic acidether a sodium compound is at first produced, analogous to the sodium acetosuccinicacidether but that this is immediately converted into the sodium salt of the acid teraconicacidether.

Lecture Experiments. V. MEYER. (Vol. XV. p. 297).

Demonstrations: Pure water is not colorless but blue. Transformation of yellow phosphorous into the red by heating in vapor of diphenylamin. Description of apparatus.

On a new Isomer of Orcin. EDM. KNECHT. (Vol. XV. p. 298).

Preparation of bioxytoluol (cresorcin) by consecutive steps from dinitrotoluol, nitrocresol, amidocresol, bioxytoluol. The new body which is a genuine homologue of resorcin (because it also has the OH groups in the meta position) is easily mistaken for resorcin. It differs from resorcin in its crystallographic forms. The author proposes the name cresorcin in order to point out its derivation from cresol and at the same time its great similarity to resorcin.

On Phosphorouspentasulphide. H. GOLDSCHMIDT. (Vol. XV., p. 303.) Determination of boiling paint by means of Meyer's and Goldschmidt's air thermometer. Mean boiling point for barometric pressure 728.5-734 mm., was found to be 518°C.

On Naphtylsulphuric Acid. R. WIETZKI. (Vol. XV., p. 305.)

It has of late repeatedly been observed that the naphthols in

*J. Bredt. Ann. Chem. 208.58.

reference to the ease with which their O H groups can be replaced, are more similar to the alcohols of the fatty series than to the phenols. Pulverized and dried naphthol rubbed in a mortar with 1½ to 2 parts of common conc. sulphuric acid at first forms a thick syrupy mass, no perceptible rise of temperature being observed. This takes place, however, after a time, and the mass crystallizes. The mass dissolved in water, neutralized with soda and a larger quantity of salt added, separates fine colorless crystals. From the reactions and characteristics of this body it is shown that it is analogous to the class of bodies known as ethersulphuric acids. The author states that an ethersulphuric acid of alpha-naphthol also exists, but he has not been able to isolate its salts. These acids cannot be combined as such with diazo compounds.

On the Capronic Acid occurring in Resin Oil. WERNER KELBE and CONSTANTIN WARTH. (Vol. XV., p. 308.)

Capronic acid was obtained from the mixture of acids occurring in resin oil by fractional distillation of the ethers of these acids. The fractions were taken at 150°-155°C. After saponifying the ether with caustic potash, the capronic acid was liberated by means of hydrochloric acid, and the free capronic acid was driven off with steam. The calcium salt was used for purification. The author finds from the properties and reactions of this capronic acid that it is identical with methylpropylacetic acid:

$$\begin{matrix} CH_3 \\ C_3H_7 \end{matrix} \Big> CH—COOH.$$

On the Action of Pentachloride of Phosphorus on Alpha-Naphtholsulphonic Acid. AD. CLAUS and H. OEHLER. (Vol. XV., p. 312.)

The action of pentachloride of phosphorus upon the sulphonic acid of alpha-naphthol differs from that upon beta-naphtholsulphonic acid in as much as the formation does not take place by ether-like combinations between the alpha-naphtholhydroxyl and sulphonic groups or from phosphoric acid ethers. Below 100° alpha-naphtholsulphonic chloride is formed. If the mixture of the alpha-naphtholsulphonic salt and pentachloride of phosphorus is heated to 100° or 150°, two new bodies—chlornaphthol and dichlornaphthaline can be isolated.

Synthesis of the Homologues of Anilin from Bromanilines, Bromtoluidines, etc., by the action of Sodium and

Alkylbromides in Ethereal Solution. AD. CLAUS. (Vol. XV., p. 315).

Xylidine from Bromtoluidine. Bromparatoluidine was employed. The effect of sodium on a mixture of this body with an excess of methyliodide in ethereal solution takes place without the formation of much pitch like substance. The oil obtained by the usual method of acidifying with hydrochloric acid, and liberating the base with caustic potash, was found not to be pure xylidine. Azotoluol and toluidin are formed in small quantities. The base or bases obtained by the same process from normal propylbromide and parabromaniline remain to be experimented upon.

On Dichlornaphtralin and Chlornaphthol from Beta-Naphtholsulphonic Acid. AD. CLAUS and M. DEHNE. (Vol. XV., p. 319).

The authors conclude as to the difference between alpha and beta naphtholsulphonic acid as follows: the tendency of the former to form basic salts is scarcely at all shared by the latter; while from the first by means of nitric acid,dinitronaphthol can easily be formed, the latter does *not* yield sulphuric acid when boiled with nitric acid, but apparently produces a new *nitrated* sulphonic acid. By treating with chlorate of potash and hydrochloric acid the alpha-naphtholsulphonic acid is converted into dichlornaphthachinon in the cold, while the same reagent acts upon beta-naphthosulphonic acid only at boiling heat, and then but very slowly.

On the Quantity of Furfurol contained in Fermented Liquids. K. FOERSTER. (Vol. XV., p. 322).

Small quantities were found in the distillates of beer and pure wines. It was also found in the liquid filtered, but not distilled from a fermented solution of 300 grains of starch-sugar (artificial dextrose). The author sums up as follows:

1. That all conclusions on the presence of amylic alcohol by the aid of Jorissen's reaction are wrong.

2. That no means have yet been found by which small quantities of amylic alcohol can be quantitatively determined with safety in fermented solutions.

3. That all suspicions referring to the use of starch sugar for improving the wine because the quantity of fusel oil was found larger, are either uncertain or false.

Benzol, Dipropargyl and Acetylene. Constitution of Benzol. JUL. THOMSEN. (Vol. XV., p, 328).

Experiments in thermo-chemistry. Difference in heat of combustion :

Dipropargyl.....	883230c
Benzol.....	787950c
Difference.....	95280c
3 molecules of Acetylene.....	931350c
1 molecule of Benzol.....	787950c
Difference.....	143400c

Author holds that this is an argument for the assertion that the carbon atoms of benzol are connected with each other by nine single bands, and not, as the customary hypothesis requires, by three single and three double bends.

On Carbostyril. P. FRIEDLANDER and H. OSTERMAIER. (Vol. XV., p. 362). Continued from Vol. XIV., p. 1916.

On the Bodies Produced from Sulpho-Carbanilid by Adition of Iodine Alkalies. W. WILL. (Vol. XV., p. 338.

Description of the methods of preparation of several of these products. The investigation is still incomplete.

On Anthracylamin. H. ROEMER. (Vol. XV., p. 223).

From amido-anthrachinon, hydroiodic acid and red phosphorus.

Instead of using litmus to indicate the limit of saturation of phosphoric acid by soluble bases, "Orange No. 3" of Poirrier (Azodemiethylamide), and "Helianthine" have been employed. The aqueous solution of these substances is yellow, but it turns red when in contact with free acids only, as demonstrated by W. Miller for "Tropeoline" (Potassium Phenylamidoazophenylsulphite).

Abstracts from the *Comptes Rendus*, By A. Bourgougnon.

Magnesium Oxychlorides. BY G. ANDRE. (No. 7, Feb. 13th, 1882).

Prepared by heating 500 grammes of water, 400 gms. of crystalized magnesium chloride, and adding to the mass when boiling 20 grs. calcined magnesia. A clear liquid is separated by filtration,

and after some time a deposit having the appearance of fine needles is formed, having the composition Mg. Cl_2, Mg. O, 16 H_2 O. Dried in vacuo until a constant weight is obtained, it gives Mg. Cl_2 MgO. 6 H_2 O.

These two oxychlorides are immediately decomposed by water or alcohol.

A New Oxychloride of Sulphur. By J. Ogier. (No. 7, Feb. 13th, 1882).

This new oxychloride has been prepared by treating in strong sealed tubes a mixture of equal weights of sulphur chloride and sulfuryl chloride. Boiling point 60°—61° C. Very easily decomposed by heat. Composition S_2 O Cl_2. Its formation is represented by the equation

$$2\,S_2Cl + 2\,S\,O_2\,Cl = 2\,S_2OCl_2 + SO_4 + S.$$

Vapor density with Meyer apparatus at 100° C. 3.28; 3.84; 3.75. Dumas' method at 60° C., under a pressure of 10 cc. of mercury gives 3.9.

Action of Potassium Cyanide upon Potassium Trichloracetate. By E. Bourgoin. (No. 7, Feb. 13th, 1882).

The action of potassium cyanide upon a concentrated solution of potassium trichloracetate is similar to the action of an alkali upon the trichloracetate.

$$C_4\,K\,Cl_3\,O_4 + K\,H\,O_2 = C_2\,K_2\,O_6 + C_2H\,Cl_3$$

Heat Developed During the Formation of Ferricyanhydric Acid. By M. Joannis. (No. 7, Feb 13th, 1882).

(The articles upon Thermochemistry do not admit of abstraction).

On Galactin. By A. Muntz. (No. 7, Feb. 13th, 1882). Extracted from the seeds of Lucerne (Medicago arborea L.)

White, translucent mass, swelling and slowly dissolved by water, giving a limpid solution; is not precipitated by neutral lead acetate, but by basic acetate. Same composition as the gums. Dextrogyrate. Treated with nitric acid, gives mucic acid.

Aconitates. By E. Guinochet. (No. 7, Feb. 13th, 1882).

(This article does admit of abstraction.)

Double Salts of Mercury. (Thermochemistry.) By M. Berthelot. (No. 8, Feb. 20th, 1882).

On Ferrocyanhydric Acid. (Thermochemistry). By M. Joannis. (No. 8, Feb. 20th, 1882).

Upon the Saturation of Phosphoric Acid by Bases and upon Chemical Neutrality. By A. Joly. (No. 8, Feb. 20th, 1882.)

Ferricyanhydric Acid. By M. Joannis. (No. 8 Feb. 20, 1882). (Thermochemistry.)

Action of Iodine upon Naphthaline at a High Temperature. By A. Bleunard and G. Vrau. (No. 8 Feb. 20, 1882).

Naphthaline is dehydrogenized by iodine; hydriodic acid is formed, and a new compound $(C_{10}H_6)_x$ has beeu obtained, which represents naphtaline less two atoms of hydrogen.

Double Salts of Mercury. By M. Berthelot. (No. 9 Feb. 27, 1882). (Thermochemistry.)

Analysis of a Volcanic Ash from Mt. Etna. By L. Ricciardi. (No. 9 Feb. 27, 1882).

Silica	37.82
Sulphuric acid	20.57
Alumina	9.97
Protonide of iron	14.05
Lime	11.98
Magnesia	3.64
Chlorine	1.02
Soda and Potassa	0.95
	100.00

Normal Carbonic Acid of the Atmosphere. By M. Dumas. (No. 10 March 6, 1882.

Among the causes which concur to equilibrate the quantity of carbonic acid in the atmosphere, the author relates an explanation given lately by Schlosing, and derived from the principle of dissociation. The proportion of carbonic acid contained in the atmosphere is in proportion with the quantity of calcium bi-carbonate dissolved in the waters of the seas. When the quantity of carbonic acid decreases, the marine calcium bi-carbonate is dissociated; half of its carbonic acid gas goes in the atmosphere and neutral calcium carbonate is deposited. When the aqueous vapor is condensed in the atmosphere, it now dissolves a portion of the carbonic acid present, and when the rain, so charged with carbonic acid, comes in contact with the earth it dissolves lime, calcium bi-carbonate is formed and carried to the sea.

The variation of the proportion of carbonic acid in the atmosphere is very small; this variation is about $\frac{3}{10000}$ in volume.

Upon the Double Decompositions of the Haloid Salts of Mercury by the Hydracids and the Haloids Salts of Potassium. By M. BERTHELOT. (No. 10 March 6, 1882). (Thermochemistry.)

The formation of two Dibasic Acids, Sebacic and Suberic During the Distillation of Crude Fatty Acid in a Current of Superheated Steam. By A. CAHOURS AND E. DEMARCY. (No. 10 March 6, 1882).

The distillation of crude fatty acids in a current of superheated steam transforms these acids into saturated hydrocarbons, homologous with marsh gas, in acids of the acetic group belonging to the normal series, and finally into two dibasic acids, suberic and sebacic acids, homologous with succinic acid.

Double Decompositions of the Haloid Salts of Mercury. By M. BERTHELOT. (No. 11 March 13, 1882). (Thermochemistry).

Crystallized Oxychloride of Gallium. By L. DE BOISBAUDRAN. (No. 11 Mar. 13th, 1882). After three years a sample of gelatinous hydrated chloride of gallium was transformed in small crystals surrounded by a liquid. The analysis of the crystals (octahedrons) has been given. $(Ga_2 Cl_6 + 12 H_2 O) + 2 (Ga_2 O_3 + H_2 O)$.

Heat of Formation of Ferricyanhydric Acid, and of some Ferrocyanides. By M. JOANNIS. (No. 11. Mar. 13th, 1882). (Thermo-chemistry).

Products of Distillation of Colophony. By A. RENARD. (No. 11. Mar. 13th, 1882). The distillate of colophony boiling at about 150° C. contains three hydrocarbons, a terebenthen $C_{10} H_{16}$ and two isomeric hydrocarbons $C_{10} H_{18}$. These three hydrocarbons can not be separated by distillation; their mixture is laevogyrate.

Dichloride of Camphor. By P. CAZENEUVE. (No. 11. Mar. 13th 1882.) Obtained by the action of dry chlorine upon camphor, dissolved in absolute alcohol. Corresponds to $C_{10} H_{14} Cl_2 O$.

Essential Oil of Licari Kanali. By H. MORIN. (No. 11. Mar. 13th, 1882).

Determination of Tannic and Oenogallic Acid in Wine. By F. JEAN. (No. 11. Mar. 13th, 1882).

Gastric Digestion. By E. Duclaux. (No. 11. Mar. 13th, 1882).

Double Decompositions of the Haloids Salts of Mercury. Conclusions. By M. Berthelot. (No. 12. Mar. 20th, 2882) (Thermo-chemistry).

New Combinations of Nitric Acid and Acetic Acid with Ammonia. By L. Troost. (No. 12. Mar. 20, 1882). By the action of dry ammonia upon dry nitrate of ammonium two nitrates were obtained.

$$N_2O_5\ HO,\ NH_3 + \tfrac{3}{2}\ NH_3$$
$$\text{and } N_2O_5\ HO,\ NH_3 + 3NH_3$$

The new combinations of acetic acid and ammonia are

$$C_2H_4O_2\ NH_3 + 3\ NH_3$$
$$\text{and } C_2H_4O_2\ NH_3 + 6\ NH_3$$

Action of Acid Solutions upon Protoxide of Iron. By A. Ditte. (No. 12. Mar. 20th, 1882).

Action of Ozone upon the Salts Manganese. By M. Maquenne. (No. 12. Mar. 20th, 1882). Ozone produces in the solutions of salts of protoxide of manganese a brown precipitate of hydrated oxide of manganese. In acidifying the solutions, other products, such as permanganic acid, are formed.

Heat of Formation Sulphocyanic Acid and of some Sulphocantes. By M. Joannis. (No. 12. Mar. 20th, 1882). (Thermo-Chemistry).

Clarification of Champagne Wines. By F. Jean. (No. 12. Mar. 20th, 1882). Tannic acid in solution in the must is titrated with a solution of iodine. When the amount of tannic acid is known an equivalent quantity of gelatine is added, the gelatine and tannic acid form with albuminoid matter an insoluble precipitate —82.8 of tannic acid requires 100 of gelatine.

Kola Nut, Seeds of Sterculia Acuminata. By E. Heckel and F. Schlagdenhauffer. (No. 12. March 20, 1882.)

Caffein	2.348	Soluble in Chloroform.
Theobromin	0.023	
Tannic acid	0.027	
Fat	0.585	

Tannic acid	1.591	Soluble in Alcohol.
Coloring matter, (red)	1.290	
Glucose	2,875	
Salts	0.070	
Starch	33.754	
Gum	3.040	
Coloring matter	2.561	
Proteids	6.761	
Ashes	3.325	
Water	11.919	
Cellulose	29.831	
Total	100,000	

Double Decompositions of Haloid Salts of Silver. By M. BERTHELOT. (No. 13, March 27th, 1882), (Thermochemistry).

Researches Upon Ozone. By M. MAILFERT. (No. 13. March 27th, 1882). The object of these researches is the action of Ozone upon organic matter, and upon several oxides and metallic sulphides, and also upon salts from which the bases can be suboxodized.

Action of Alkaline Solutions Upon Protoxide of Tin. By A. DITTE. (No. 13. March 27th, 1882.)

Upon Campholurethane. By A. HALLER. (No. 13. March 27th, 1882.)

Preparation of Pure Carbon for Electric Light. By M. JACQUELAIN. (No. 12. March 27th, 1882.) The impurities in carbon are removed by treatment with chlorine, caustic soda or hydrofluoric acid, and the carbon is then washed in water.

Haloids Salts of Silver and Potassium. By M. BERTHELOT. (No. 14. April 3d, 1882).

Union of Free Hydrogen with Ethylen. By M. BERTHELOT. (No. 14. April 3d, 1882).

The combination is limited by the dissociation of the hydrides, and the degree of dissociation varies rapidly with the temperature.

Effects of Compression upon the Hardness of Steel. By M. LAN. (No. 14. April 3d, 1882).

Steel is hardened by compression. In compressed steel the pro-

portion of combined carbon is increased and the proportion of free carbon diminished, as shown by the following determinations:

	Compressed.		Non-Compressed.	
Carbon, total,	0.70 per ct.		0.70	
Combined carbon, A,	0.60 "	Average 0.585	0.49	Average 0.490
" " B,	0.59 "		0.50	
" " C,	0.55 "		0.47	
" " D,	0.60 "		0.50	
Free carbon (by difference)	0.115		0.21	

Upon the Composition of Hydrated Carbonic Acid. By M. WROBLENSKI. (No. 14. April 3d, 1882).

At 0°C and under the pressure of about 16atm. hydrated carbonic acid has the composition $CO_2 8H_2O$.

Synthesis of Quinine. By E. G. MAUMENE. (No. 14. April 3d, 1882).

The author states that having discovered the compound NH_2 he has been able to prepare artificially some very pure sulphate of quinine, but that before describing his process he will make some comparative experiments in order to test the medical properties of this artificially prepared quinine.

Action of Fuming Nitric Acid Hydrochloric Acid, upon Pilocarpin. By P. CHASTAING. (No. 14. April 3d, 1882).

Fuming nitric acid transforms pilocarpin into nitrate of Jaborandin $C_{20}H_{12}N_2O_6NHO_6$.

By the action of hydrochloric acid pilocarpin is transformed into chloride of jaborandin.

Transformation of Carbon Oxysulphide into Ordinary and Sulphurretted Ureas. By M. BERTHELOT. (No. 16. April 17th, 1882).

Chemical Monography of the Cucurbitaces of Uruguay. By F. SACC. No. 16. (April 16th, 1882).

The author gives several analyses of the melons coming to the Montevideo market, also the analysis of the roots of Briony, which are often mistaken for wild potatoes. They have the following composition:

Pectic acid	0.33 per cent.
Lignine	9.65 "
Inulin	5.32 "
Grape sugar	4.86 "
Water and loss	79.84 "
	100.00

Separation of Gallium. By M. LECOQ DE BOISBAUDRAN. (No. 17. April 24th, 1882).

Upon the Atomic Weight of Carbon by the Combustion of Diamond. By H. E. ROSCOE. (No. 17. April 24th, 1882).

African diamonds were employed. They did not yield a trace of hydrogen. Average of six experiments 11.07, oxygen being 15.96.

Decomposition of Lead Salts by Alkalies. By A. DITTE. (No. 17. April 24th, 1882).

Researches upon Ozone. By M. MAILFERT. (No. 17. April 24th, 1882.)

A study of the action of ozone upon sulphur, selenium, tellurium, sulphides and some organic matters.

Insoluble Modification of Pepsine. By A. GAUTIER. No. 17. April 24th, 1882).

Oxydation of Pyrogallol in the Presence of Gum Arabic. BY P. de CLERMONT AND P. CHAUTARD. No. 18. (May 1st, 1882). The produce of oxidation is Purpurogallin, having the appearance of yellow needles C_{20} H_{16} O_9.

Chemical Studies of Several Products of Uruguay. BY F. SACC. No. 18. (May 1st, 1882).

Composition and Atomic Weight of Pernitric Acid. BY P. HAUTEFEUILLE AND G. CHAPIUS. No. 19. (May 8th, 1882).

Action of Potassa Upon Lead Oxide. BY A. DITTE. No. 19. (May 8th, 1882).

New Compounds of Carbon and Silicium. BY A. COLSON. No. 19. (May 8th, 1882).

This new compound is made by means of a stream of hydrogen saturated with benzol, passing over silicium placed in a red-hot porcelain tube.

Upon the Homologous and Isomeric Rosanilin. BY. A. ROSENSTIEHL AND M. GERBER. No. 19. (May 8th, 1882).

Composition of Ashes from Vesuvius. BY L. RICCIARDI. No. 19. (May 8th, 1882).

Eruption of February 25th, 1882.

Silica		47.84
Anhydr.	Sulphuric Acid	0.17
"	Phosphoric Acid	1.83

Chlorine	1.32
Alumina	18.67
Protoxide of Iron	5.07
Peroxide of "	4.38
Lime	9.42
Magnesia	3.77
Potassa	5.64
Soda	2.04
	100.15

Studies of the Antiseptic Properties of Salicylic Acid. By E. Robinet and H. Pellet. No. 19. (May 8th, 1882).

Upon Some Reactions of Mercuric Chloride. By H. Debray. No. 18. (May 1st, 1882).

Mercuric chloride is reduced to mercurous chloride by the solution of sulphurous acid, the presence of sodium chloride prevents the reduction.

When a solution of an alkali is mixed slowly with a solution of mercuric chloride, the precipitate varies in color from yellow to black, especially in warm liquids, these different colorations are produced by the formation of oxychlorides of variable composition. With an excess of alkali, all the oxychlorides are destroyed, and the precipitate is formed only of yellow oxide of mercury. The presence of sodium chloride prevents the formation of these intermediary products.

Liquefied Gases, and Particularly Ethylen, for the Production of Low Temperatures. By L. Cailletet. No. 18. (May 1st, 1882).

The evaporation of ethylen in an open vessel produces a temperature of—105° C.; this temperature is far lower than that produced by nitrous oxide which boils at—88° C.

Separation of Gallium. By M. Lecoq de Boisbaudran. No. 18. (May 1st, 1882).

Liquefaction of Ozone. By P. Hautefeuille and G. Chapuis. No. 18. (May 1st, 1882).

Drops of liquefied ozone had been obtained with a Cailletet's apparatus, the liquid is dark-blue indigo, and had been preserved for thirty minutes under a pressure of 75 atm. Its vaporization is

not very rapid even under atmospheric pressure. The liquefaction has been obtained by compressing a mixture of oxygen and ozone under 125 atm., the refrigerating liquid being ethylen, and the temperature was below—100 C°.

Action of Insoluble Metallic Sulphides upon a Solution of Acid Sulphate of Nickel in the presence of H_2S. By. H. Baubigny. No. 18. (May 1st, 1882).

Synthesis of Organic Compounds by Means of Electrolysis of Water, Acid, Alkaline and Alcoholic Solution with Carbon Electrodes. By A. Bartoli and G. Papasogli. No. 20. (May 15, 1882).

On the Absorption of Gases by Platinum. By M. Berthelot. No. 21. (May 22, 1882).

Action of Peroxide of Hydrogen upon Organic Matter and Fermentation. By P. Bert and P. Regnard. No. 21. (May 22, 1882). Very diluted peroxide of hydrogen stops the fermentations produced by living organisms and the putrefaction of all substances which do not decompose it. It has no action upon the diastasic fermentations. Diluted peroxide of hydrogen is not destroyed by fats, amylaceous matter, soluble ferments, egg albumen, casein, peptones, creatin,creatinin, or urea,but is quickly decomposed by musculin, fibrin and generally by vegetable nitrogenized matter.

Separation of Gallium. By Lecoq de Boisbaudran. No. 22. (May 29, 1882). Analytical methods for the separation of gallium from glucinium, cerium, lanthanum, didymium, samarium, yttrium, erbium, holmium, thalium, iron and thorium.

Determination of Glycerin in Fatty Matters. By J. David. No. 22. (May 29, 1882). The saponification is made with crystals of barium hydrate, alcohol and water; alcohol is separated by boiling and barium with suphuric acid. The clear liquid is evaporated to a determined volume, its sp. gr. taken and compared with the sp. gr. of a solution of glycerin and water of a known composition.

Double Salts Prepared by Fusion. By MM. Berthelot and Flosray. No. 23. (June 5, 1882).

Oxychloride of Zinc. By G. Andre. No. 23. (June 5, 1882). ⅓A new oxychloride of zinc. 2 ZnCl 5 Zn_2O 26H_2O is described.

Action of Carbon Di-Sulphide upon Silicium. By A. Colson. No. 23. (June 5, 1882).

Preliminary Note on Didymium. By T. Cleve. No. 23. (June 5, 1882). The differences obtained in the determinations of the atomic weight of didymium have induced the author to believe in the existence of a new element which he calls beta-didymium.

Explosible Alloys of Zinc with the Platinum Metals. By Sainte-Claire Deville and H. Debray. No. 24. (June 12, 1882).

Separation of Gallium. By Lecoq de Boisbaudran. No. 25. (June 19, 1882). Separation of gallium fron zirconium, manganese and zinc.

Upon Silicium. By P. Schutzenberger and A. Colson. No. 26. (June 26, 1882).

On the Compound NH_2. By M. Combes. No. 26. June 26. 1882). According to the author, the compound NH_2 has not been obtained. NH_3 and carbonic acid had been formed in the reaction alluded to by M. Maumene.

On Didymium. By B. Brauner. No. 26. (June 26, 1882). Ordinary didymium is a mixture of three elements—didymium, beta-didymium and perhaps samarium.

Action of Peroxide of Hydrogen upon the Coloring Matter of Blood and upon Hematosin. By A. Bechamp. No. 26. (June 26, 1882). Description of the action of peroxide of hydrogen upon hemoglobin and hematosin and upon the white matter of hemoglobin.

Electrolysis of Hydrogen Dioxide. By M. Berthelot. (No. 1, July 3d, 1882.)

Remarks on Didymium. By P. T. Cleves. (No. 1, July 3d, 1882.) In a preliminary note the author suggested the existence of a new element which he named Beta-Didymium, but further experiments have shown the non-existence of this new element.

Action of Hydrogen Sulphide upon Nickel Chloride. By H. Baubigny. (No. 1, July 3d, 1882.) The transformation into sulphides of some metallic chlorides is more difficult than the transformation of the corresponding sulphates, under the same conditions,

by the action of hydrogen sulphide. Equivalent weights of sulphate and chloride of nickel being taken and submitted under the same conditions to the action of hydrogen sulphide, it has been observed that the conversion of the sulphate into sulphide was quicker than for the chloride.

Isomery of Cuprous Sulphites. By M. ETARD. (No. 1, July 3d, 1882.)

Reduction of Some Silver Ores by Hydrogen and the Wet Way. By P. LAUR. (No. 1, July 3d, 1882.) The silver ore, sulphide, chloride, iodide or bromide in fine powder is placed in a cast iron pan with a weak alkaline solution (1 part of soda for 100 of water) and an amalgam of 3 parts of tin for 100 of mercury and submitted to ebullition, nascent hydrogen decomposes the argentiferous compounds, the silver forms an amalgams, and the sulphur is transformed into an alkaline sulphostannate, chlorine, bromine and iodine give corresponding soda salts.

Action of Chloroform on Beta-Naphtol. By G. ROUSSEAU. (No. 1, July 3d, 1882.) By the action of chloroform upon beta naphtol several products are formed which can be separated into two distinct groups. 1 Insoluble in alkalies. 1 Glycol $C_{22}H_{14}O_2$. 2. Ether derived of the glycol $C_{22}H_{12}O$. 3. A monoatomic alcohol $C_{22}H_{14}O$. 4. A rosin containing 96 per cent. of carbon. 2d. Soluble in alkalies.—1. Aldelyde $C_{11}H_8O_2$. 2. A rosin containing oxygen. During the reaction carbon monoxide is continually evolved.

Extraction of Vanadium from the Basic Slags of "Le Creuzot." By G. WITZ and F. OSMOND. (No. 1, July 3d, 1882.) These slags have the following composition:

Silica	16.50		
Alumina	3.80		
Lime	46.30		
Magnesia	4.00		
Protoxide of iron	7.07	Iron	5.50
" manganese	5.30	Manganese	4.10
Sulphuric acid	0.63	Sulphur	0.25
Phosphoric "	13.74	Phosphorus	6.00
Vanadic "	1.92	Vanadium	1.08
	99.26		

In the metallurgical establishments of Le Creuzot alone about 60,000 kilogr. of vanadium are annually produced in the slags, and it can be extracted as ammonium metavanadate or transformed

into new products especially adapted for the fabrication of aniline black.

Basic Salts of Manganese. By A. GORGEN. (No, 2, July 10th, 1882.)

Action of Bromine upon Quinolein and Pyridin. By E. GRIMAUN. (No. 2, July 10th, 1882.) By the action of bromine upon quinolein a bromide C_9HNB^2 is produced which is very instable and easily converted into $C_9H_7NB^2$, H Br. Similar products are obtained by the action of bromine upon pyridin.

Upon the Presence of Glycol in Wine. By A. HENNINGER. (No. 2, July 10th, 1882.) Glycol is one of the products formed during the vinous fermentation.

Analyses of Water from the Isthmus of Panama. By M. AILLAUD. (No. 2, July 10th, 1882.) Analyses of well water and of the river Rio Grande taken to the south and near Panama.

Hydrate of Hydrogen Sulphide. By M. DE FORCRAND, (No. 3, July 17th, 1882.) A claim of priority.

On the Gastric Juice. By P. CHAPOTREAUT. (No. 3, July 17th, 1882.) The aqueous solution of gastric juice acidified with sulphuric acid deposits and white pulverulent mass which is considered by the author as the active principle of gastric juice, it is insoluble in an excess of sulphuric acid, but soluble in hydrochloric acid. Its percentage of carbon, hydrogen and nitrogen is:

C	51
H	7.2
N	15.4

This white precipitate transforms meat into peptone.

Two New Antiseptics. By G. LEBON. (No. 3, July 11th, 1882.) Calcium and sodium, Glyceroborates.

Separation of Gallium. By LECOG DE BOISBAUERAU. (No. 4, July 24th, 1882.) Description of methods for the separation of gallium from cobalt, nickel, thallium.

Vaporisation of Metals in Vacuo. By E. DEMARCAY. (No. 4. July 24th, 1882.)

Determination of Astringent Matters in Wine. By A. GIRARE. (No. 4, July 24th, 1882.)

PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

Room 1, *University Building, N. Y.*

The last regular meeting was held Friday, Nov. 3d.

The meeting was called to order at 8:40 P. M., Prof. A. R. Leeds in the chair.

A quorum not being present, the first paper of the evening, "On the action of Phthalic anhydride, upon Gallic acid," by Mr. James H. Stebbins, Jr., was then read, at the conclusion of which Dr. Prochazka remarked that he had produced galleine by the above method, over a year ago.

After which the second paper, "On the preparation of Asbetos, for filtering purposes," by Mr. Casamajor followed.

A quorum being now present, the minutes of previous meeting were read, and approved.

The minutes of the Board of Directors were then read, and after a correction, approved.

Dr. Doremus then stated on behalf of the Committee on Papers and Publications, that the October number of our journal is now in print, and will probably be issued next week.

He further laid before the society the necessity for members to send in more original MS., as thereon depends the success of our journal.

The committee on Endowment Fund had no report.

The following gentlemen were then elected:

Cuthbert P. Conrad, Active Member. { J. W. Mallett, E. Waller, James H. Stebbins, Jr.

Edwin V. Wilmowski, Regular Member. { James H. Stebbins, Jr., Wm. Ruppp, Dr. Grothe.

Alfred L. Beebe, Regular Member. { J. B. Macintosh, A. L. Colby, A. H. Elliott.

John Cawley, Regular Member. { James H. Stebbins, Jr., A. H. Elliott, P. Casamajor.

N. Hathaway, Regular Member. { E. Waller, Wm. Rupp, C E. Munsell.

Geo. E. Leguin, Associate Member. { Nelson H. Larton, James H. Stebbins, Jr., Wm. Rupp.

John Grey Macfarlan. { P. Casamajor, James H. Stebbins, Jr., A. H. Elliott.

The following gentleman was nominated as a Regular Member:

Percy Neymann. { E. Waller, A. H. Elliott, A. P. Hallock, } Proposed by.

The election of candidates for the Committee on Nominations was now in order.

The following gentlemen received the largest number of ballots:

1. Arthur H. Elliott.
2. E. Waller.
3. James H. Stebbins, Jr.
4. A. R. Ledoux.
5. Wm. Habirshaw.
6. P. Casamajor.
7. J. P. Battershall.
8. A. P. Hallock.
9. O. Grothe.

Three names out of the nine were then drawn from a hat by the Chairman.

The members of the Nominating Committee are therefore,

Wm. Habirshaw, J. P. Battershall, P. Casamajor.

There being no further business, the meeting adjourned.

JAMES H. STEBBINS, Jr.,
Recording Secretary.

ON THE ACTION OF PHTHALIC ANHYDRIDE, UPON GALLIC ACID.

BY JAMES H. STEBBINS JR., A.B., F.C. S.

In 1871 Baeyer observed, while heating a mixture of phthalic acid, and pyrogallic acid, the formation of a coloring matter,* which he termed "galleine."

At first Baeyer, believed that the formation of this dyestuff, was due to an oxydation of pyrogallic acid, and that the phthalic acid was not essential for its formation, as it seemed that the same dyestuff

*Ber. d. d. chem. Gesell. IV., p. 455, 555, 663.

was formed by heating pyrogallic acid, with other organic acids, or with benzaldehyde.

Further investigation, however, soon showed, that the phthalic acid, as well as the other acids, on being heated with pyrogallic acid, enter the molecule of the resulting compound, and that not only does pyrogallic acid act in this way, but also phenoles on the one hand, and many organic acids on the other, combine under elimination of water, when the mixture, is either heated alone with glycerine, or with sulphuric acid.

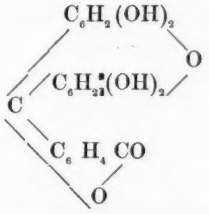
The constitution of the compounds (which he called phthaleines) was in later years explained by Baeyer, and his scholars.*

Baeyer in his last paper on this subject (2) assumes that the formula of galleine may be represented by

$$C_{20}H_{14}O_8$$

which on being heated to 180°C loses water, and passes over into a compound closely resembling fluoresceine, which he called galline. ($C_{20}H_{12}O_7$)

In order not to make this subject too tedious, I will omit the many deductions, as to the correct formula of galleine, and will use Baeyer's formula hereafter, viz.:



While reading over some of Baeyer's interesting work on this subject, it occurred to me, that possibly phthalic-anhydride, and gallic acid, might be combined, to form a compound of similar nature to galleine.

According to Baeyer's theory, phthalic-anhydride and gallic acid should unite, under elimination of water, to form a substance, which might possibly be represented by the following formulæ :

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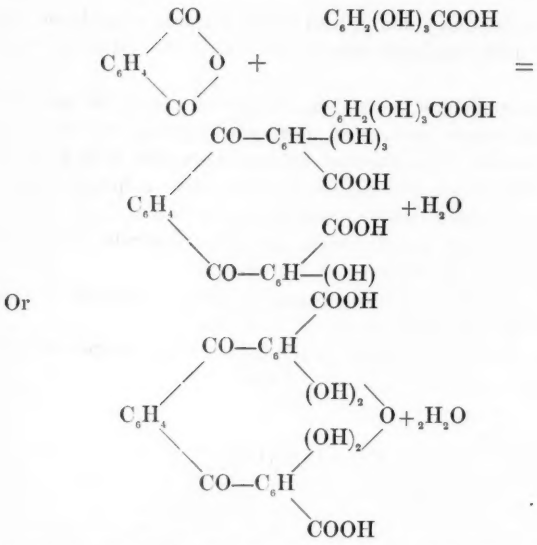
* Ann. Chem. and Pharm. 183,1; 202 p. 36.

2 Ber. d. d. chem. Gesell. IV. p. 663.



Or

But, the reaction did not work according to my expectations, as will be seen from the following:

10 grs. phthalic-anhydride, and 20 grs. gallic acid, were thoroughly mixed and heated at first, (on the sand bath) to 195°C. At this temperature no reaction took place. So the heat was gradually raised to 220°C., at which temperature the mixture melted, under evolution of a gas, which on examination was found to be CO_2. The melt then darkened in color, and finally turned red brown.

The whole time of heating was about two hours.

The melt was then allowed to cool, dissolved in boiling alcohol, and the solution so obtained poured into cold water.

A copious fine brown precipitate is thus formed. This was collected on a filter, washed with water, dried, and finally recrystallized from alcohol

A combustion of the above dried at 120°C. gave the following figures:

FOUND.	
Carbon	61.50 per cent.
Hydrogen	3.50 per cent.
THEORY.	
Carbon	62.82 per cent.
Hydrogen	3.63 per cent.

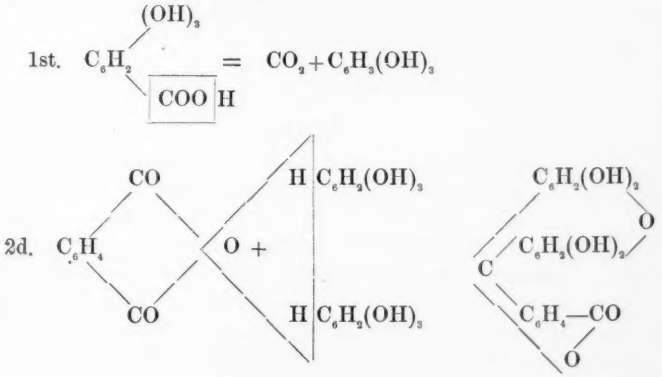
These figures, beyond a doubt, lead to the formula of galleine, and in fact the body analysed closely resembles the latter in every respect.

It dissolves in alcohol, with a fine cherry-red color. Is sparingly soluble in hot water, and dyes mordanted cotton, of the same shades as galleine. It is likewise soluble in caustic soda solution, with a fine blue color, and finally, it forms with sulphuric acid, a condensation product, closely resembling coeruleine.

The formation of galleine from phthalic-anhydride, and gallic acid may, I think, be explained as follows.

1st. The gallic acid, on being heated to 220°C. splits up into carbonic acid, and pyrogallic acid.

2nd. The pyrogallic acid on being formed then unites with the phthalic acid to produce galleine. Thus:



Finally, to make sure, that the compound under examination was really galleine, I treated a small amount of it with acetic anhydride, and thus obtained the well known tetra acetyl galleine, as may be seen from the following figures:

	FOUND.	
	I.	II.
Carbon	64.09 per cent	64.09 per cent.
Hydrogen	4.71 per cent	4.27 per cent.

THEORY.

$C_{20}H_8(OCOCH_3)_4$

Carbon	63.15 per cent.
Hydrogen	3.75 per cent.

The yield of galleine by this process is quite large, and it promises, I think, to be a good improvement in the manufacture of galleine, on account of the greater cheapness of the raw materials.

N.B. After this paper had been read before the American Chemical Society, Dr. Prochazka arose and stated that he had obtained the same results as myself, about a year previous, and I therefore cannot claim any originality in these experiments, but publish them merely for the benefit of the public, as Dr. Prochazka s experiments have remained hid under a bushel.

JAMES H. STEBBINS, JR.

NOTE ON ASBESTOS FILTERS.

BY P. CASAMAJOR.

An extensive dealer in wares used by chemists, lately informed me that he had many inquiries concerning asbestos for filtering liquids in chemical analysis. Some chemists complain that they cannot get clear solutions through asbestos, while others, who obtain clear solutions, find that their liquids filter altogether too slowly.

Allow me to recall that the method of making asbestos filters, by pouring a thin paste of this material over a perforated platinum disc, was first proposed by me in 1875,* but I neglected to give directions concerning the preparation of asbestos, to make it fit for filtering liquids in chemical analysis. It now appears, however, that such directions would be found useful, and I propose to repair the omission and give the necessary details.

The kind of asbestos to use is a matter of some importance. I have tried three kinds, which are sold by dealers in New York, as the *Canadian*, the *Italian*, and the *Australian*. This last is less flexible than the other two, and consequently the fibres do not felt together and pack as closely on the perforated plate. Hence, liquids filter more rapidly, and the Australian is, on this account, preferable to the other two kinds. I am informed that the Canadian asbestos is the most soluble in acids, but I have not verified the assertion.

Whatever may be the kind of asbestos used, the following is a

* See *American Chemist*, V., p. 44. *Chemical News*, XXXII., p. 46.

process for obtaining, with little trouble, a quantity of the pulp in a fit state for filtration:

A coarse brass sieve is placed over a sheet of paper, and a handful of asbestos is rubbed pretty roughly over the sieve cloth. This breaks it up in such a way, that the smaller fragments pass through the meshes, and are deposited on the paper underneath. After a while, the portion which remains on the sieve-cloth is collected in one bundle, and rubbed again in the same manner, and the operation is repeated until a sufficient quantity has gone through. In a few minutes, enough of the material is obtained to last for months.

As to the coarseness of mesh to use, I may say that I have used No. 10 sieve (ten openings to the inch) with satisfactory results. The sieve is best placed bottom up, so as to leave plenty of room under the cloth.

The next operation is to free the sifted material from dust and from the finest particles. This is easily accomplished by placing the asbestos, as obtained above, over another sieve of finer mesh (about No. 25 or No. 30), and stirring it while water is poured over the sieve. The first water which passes through is quite milky, but it gradually becomes clearer as the washing is continued. The washed asbestos is then put in a beaker glass, and boiled for about half an hour with strong hydrochloric acid (about 1 part of fuming HCl. to 4 parts of water).

The pulp, after this treatment, is poured over a perforated platinum plate placed in a funnel,* and washed with distilled water until no acidity is shown by litmus paper. The pulp is then taken out of the funnel and strongly heated in a platinum dish. After letting it cool sufficiently, it may be placed in a wide-mouth bottle for future use.

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* A perforated platinum disc, having a stout platinum wire soldered with gold in the centre of the disc, can be used in an ordinary conical funnel. The wire finds its place in the stem of the funnel, and keeps the perforated plate in position. (See *Chemical News*, XLVI., p. 8). A Gooch crucible of sufficient size may also be used for the same purpose.

FAT EXTRACTING APPARATUS.

T. O'Conor Sloane, Ph.D.

In many processes of analysis a continuously working apparatus for extraction with alcohol or ether is useful. The extraction of chinchona barks, for separation of the alkaloids may be instanced. In their analysis it is best to work upon large quantities, and a long treatment with the solvent is necessary. Any attempt to thoroughly extract a large quantity by percolation and decantation would be tedious and probably imperfect. A well arranged fat-extractor is particularly available for analysis of this type.

The cut, accompanying this, shows a very efficient and cheap form of fat extractor.

A represents a glass receptacle in which the substance weighed out for analysis is placed. It will be recognized as one of the pieces used in the apparatus for extraction of soils. The bottom may be closed by a piece of sponge, and broken glass placed on this to a depth of two or three centimetres. The substance to be extracted rests on the broken glass.

B represents a glass flask. The alcohol or other extracting fluid is boiled in it, and the vapor is condensed in the condenser shown above it.

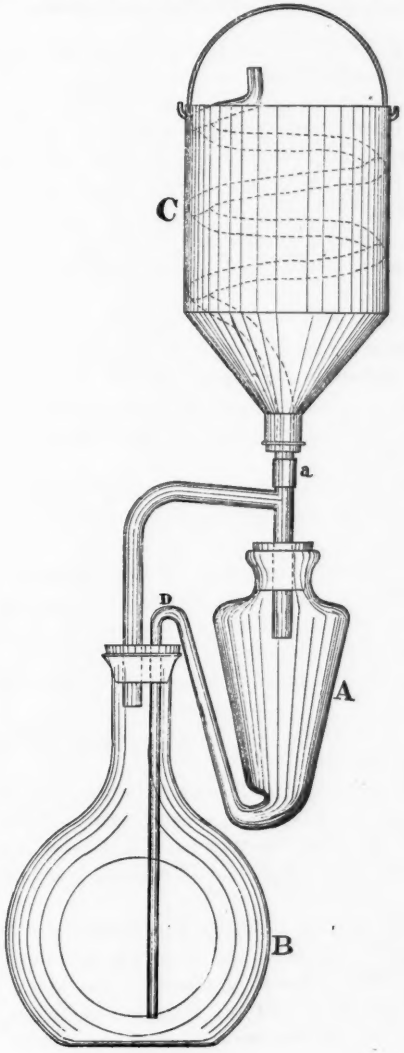
C is the condenser in question. It may be made with block tin pipe for the coil, as shown by the dotted lines, and a varnish can may serve for the case. If the varnish should be used, the bottom must be cut off, and the handle transferred from top to bottom as shown. The tin coil pipe passes through a perforated cork in the neck.

The other connections may be made of glass tubing, joined to the tin tube at *a* by a rubber tube.

The condenser is to be filled with water. While a continuous supply is desirable, it is not necessary, provided the condenser be large enough. In that case the water may be changed every hour, the warm water being syphoned off without dismounting the apparatus.

The operation is simple. The fluid in the flask *B* is boiled. Its vapor rises, condenses in the condenser *C*, and drops down into the receiver *A*. When sufficient has accumulated to rise above the bend *D* it syphons into the flask, and the fluid is rapidly drawn out of the receiver, until by the admission of air at the bottom of the receiver

the syphon is broken. A new accumulation of condensed fluid then takes place, which is soon syphoned off. Thus a continuous series



of rapid percolations is produced, exhausting the substance in a most effective manner.

With a boiling flask of a litre capacity, a receiver of four hundred c. c. capacity, and a condenser of five litres capacity, the extracting fluid being alcohol, the syphonings should follow each other at intervals of about twenty minutes.

The nearly horizontal portion of glass tube to the left of *A* should pitch downwards towards the receiver. This is very important, as otherwise the condensed fluid would partly run back into the flask.

Where alcohol or ether is used, a water bath should be employed to boil it.

All the tube connections should be as large as possible to insure rapidity of working.

ON BISULPHIDE OF CARBON.

By L. H. Friedburg, Ph. D.

Several years ago I published some notes on bisulphide of carbon* to which I shall add to day a few more observations. Then and there I showed how to clean the bisulphide by means of fuming nitric acid, and that the vapors of nitrous acid, of nitrogen dioxyde, of sulphurous acid, etc.,etc., were taken up and invariably retained by the bisulphide. Dry bisulphide of carbon serves as a very good conveyance for the reaction of such gases and vapors in a dry state on each other and on other substances. The only disagreeable feature in this regard is that carbon bisulphide in most cases also enters the reaction, forming very undesirable products, and sometimes, only such, sulphur containing, products are formed, in any notable quantity. The following reactions are the only three I wish to mention, as they may prove germs for further investigations.

1. Bisulphide of carbon charged with the vapors of nitrogen dioxyde and then mixed with pure benzol,forms amongst other products large,broad crystals of dinitro-benzol,melting at+86°C. These crystals are formed after standing a considerable time, and after the partial evaporation of the mixed liquids at summer heat.

2. I think that great interest is attached to the reaction of the aforesaid liquids in direct sunlight. The brown vapors begin to disappear without escaping from the narrow neck of the very large flask, in which such experiments take place, and in proportion as they disappear, small white crystals begin to cover the sides of the

* Berichte d.d. ch. Ges. VIII., 1616.

flask within. This covering principally takes place above the edge of the liquid on the bare glass. The crystals could not be analyzed, because they decomposed when brought in contact with air, yielding then NO_2 and benzol. It is not entirely out of the way to suppose, that in this case addition products formed, analogous to benzol-hexachloride $= C_6H_6Cl_6$, so that the white crystals in this case, might be either $C_6H_6(NO_2)_4$ or $C_6H_6(NO_2)_6$, which of course needs further investigation.

3. A very pretty reaction takes place, when bisulphide of carbon charged with dry sulphurous acid gas, and the same medium charged with nitrogen dioxyde (which was not free from nitrous acid) are brought together. This reaction might be used as a lecture experiment. Keeping the vessels cool and dry (I generally use a spacious beaker) white crystals very readily form in considerable quantity, which, in fact, are nothing but lead chamber crystals. This reaction treated analytically may some day throw new light on the formation and composition of lead chamber crystals.

The bisulphide of carbon cleaned by means of fuming nitric acid is the only chemically pure I came across, and I therefore proceeded to determine its specific gravity as well as boiling point, without finding though any differences from former determinations.

The spec.-gravity at $+$ 15°.2 C. is 1,266 and the boiling point is $+47.4$ C., at 0. 760m pressure.

The pure bisulphide shows materially no other so-called physical properties than those known heretofore.

Finally I want to state, that in treating raw bisulphide of carbon, coming from the retorts of manufacture, with fuming nitric acid, I could invariably detect mononitrobenzol in the residue of evaporation, which leads me to believe that amongst the numerous products formed in the red hot retorts, particularly if the charcoal was not dry enough, there is also benzol.

2. The purifying influence of a non-colored and inodorous fat, for instance such as the oil of African palm kernels* good as it is for the bisulphide of carbon, becomes a nuisance, when this latter is used as a means of extraction of the former. I advise, therefore, from long experience, all those who extract fats (particularly if it is for soap manufacturing) by means of CS_2 never to use an excess of this latter, and never to let a new mixture of oil and bisulphide run into the still in which oil already freed from bisulphide is retained. This latter will else be rendered impure.

*See foot note page 252.

NOTE ON THE ACTION OF ALCOHOLIC POTASH ON HEPTYLENE BROMIDE.

F. P. Venable, Ph. D.,

Having on hand a small supply of heptylene bromide, described in this journal (IV. 22), the action of an alcoholic solution of potassium hydrate was tried upon it with a view to the formation of a hydrocarbon with two less carbon atoms a heptine isomeric with the oenanthylidene of Limpricht (Liebig's Annalen 103.81) and the tetra methylallylene of Henry (Ber. Chem. Ges. 8. 400). These two bodies were formed by the action of alcoholic potash on the chlorides. It was interesting then to see if the reaction with this bromide would follow the same course.

From seven to eight grammes of heptylene bromide were placed in a flask and an excess of a strong alcoholic solution of caustic potash added (2 mol. KOH to 1 mol. $C_7H_{14}Br_2$ being the proportion required by formula). To condense and return the alcohol to the flask it was connected with an upright condenser. The reaction commenced immediately in the cold—potassium bromide beginning to deposit—but it went on very slowly without the aid of heat. The whole was accordingly heated 4–6 hours on a water bath, the potassium salt forming a thick deposit on the sides of the flask and the liquid, at first colorless, darkening to a reddish black. At the expiration of this time no further precipitation of potassium bromide was observable on adding fresh alcoholic potash. The oily liquid was thoroughly washed with water, separating in a lighter layer at the top of the separating funnel, was dried over calcium chloride and finally fractioned under 100°C., the amount coming over was small. The thermometer thé rose rapidly to 150°C. then slowly to 160°C. Between these two degrees nearly the whole product came over, leaving a dirty, thick mass behind in the fractioning flask. On redistilling the fraction from 150°–160°C, a small portion of a constant-boiling product was obtained—boiling at 156°–158°C. This was immediately sealed in a tube and set aside for analysis. On examining it qualitatively, bromine was found present in considerable amount so that clearly some brominated compound instead of the expected C_7H_{12} had been formed by the reaction. Seeing that Bruylaut (Ber. Chem. Ges. 8. 409) had prepared a monobromœnanthylene by the action of alcoholic potash on oenanthylene bromide (heated together two days in closed tubes), a

bromine determination was made after the method of Carius to see if some similar body had not been found in this case. A confirmatory determination would have been made but unfortunately part of the liquid was lost.

0.1895 grm. substance gave 0.2019 grm. silver bromide.

Calculated for $C_7H_{13}Br$, 45.19 per cent. Br—found 45.34 per cent. Probably the first small fraction coming over somewhat under 100° consisted of C_7H_{12} but the quantity was too small for examination and purification. The reaction then, instead of following the formula, $C_7H_{14}Br_2 2KOH = C_7H_{12} + KBr + 2H_2O$ was rather $C_7H_{14}Br_2 + KOH = C_7H_{13}Br + KBr + H_2O$ in spite of the excess of alcoholic potash.

Bromheptylene—$C_7H_{13}Br$ is a colorless mobile liquid, with a pleasant though pungent aromatic odor, lighter than water and boiling at 156°–158°C. The two bodies $C_7H_{13}Br$, and C_7H_{12}, prepared by Bruylaut had respectively the boiling points 165°C and 100°C. This gives a difference of 7°–9° in boiling points between the derivation from the œnanthal heptane and the Pinus Sabiniana heptane. Nearly the same difference is noticed between the heptylic bromides $C_7H_{15}Br$ from œnanthal boiling at 178.5° and from P. Sabiniana boiling at 165.5.°

University of North Carolina.

NOTES FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF NORTH CAROLINA—ANALYSIS OF ROCK-SALT FROM SALTVILLE, WASHINGTON CO., VA.

By Thomas Radcliffe.

A specimen of this rock-salt sent by the superintendent of the salt-works in the valley of the Holston, yielded on analysis somewhat different results from the previously published analysis (Chem News No. 1038) and in view of this and the claim made for this Virginia brine, that it exceeds in purity nearly all others of which analyses are on record, the analysis made in this laboratory is published. The specimen was brownish-red in color, with a crystalline structure and was obtained whilst deepening one of the salt-wells. This rock-salt is not mined, the brine alone being used for the manufacture of salt. The capacity of the works is at present 450,000

bushels per year, though at one time, during the late war, the yield was as high as 10,000 bushels per day.

According to this analysis the rock-salt contained:

Na Cl	93.05
K Cl	trace
$CaSO_4.2H_2O$	2.40
$MgSO_4$	.07
Fe_2O_3	.83
SiO_2	2.81
H_2O	.30
	99.46

An analysis of the salt as marketed gave 98.89 per cent. NaCl with a small percentage of $CaSO_4 2H_2O$ and a trace of $MgSO_4$ showing it to be a high-grade salt.

ANALYSIS OF A DEPOSIT OF ZINC OXIDE IN A BLAST FURNACE AT LONGDALE, VA.

By Thomas Radcliffe.

As is well-known these deposits of impure zinc oxide are some times found in furnaces where zinc-bearing ores are used. The name cadmia is given them in Dana's mineralogy. The green flame of burning zinc is noticed at the tyrup of these furnaces and was formerly looked upon by furnace-men as indieative of sulphur—especially as this burning left on substances in the near neighborhood of the tyrup a coating of zinc oxide which was yellow whilst hot. The specimen examined was sent through the courtesy of the manager of the Longdale Iron Co's. furnaces. According to analysis no zinc is contained in the ores used by this company, the said ores being ordinary brown hematite. Nor has zinc been found in the coke and limestone used—evidently occurring then in minute traces probably in the ore. The deposit was very large, nearly choking the mouth of the furnace. The specimen had a laminated appearance, as if deposited in layers, and was greenish-brown in color. It was quite hard, breaking in thin plates, like shale, in the

direction of the lamination and the specific gravity was 5.0405 (temp. of water 16°C).

The analysis gave:

ZnO	93.34
PbO	2.37
Fe (metallic)	1.99
$CaCO_3$	1.01
C	.20
SiO_2	1.11
	100.02

ABSTRACTS.

Abstracts from the Journal of the Chemical Society (London), by Arthur H. Elliott, Ph. B., F. C. S.

On Rotary Polarization of Chemical Substances under Magnetic Influence. By W. H. PERKIN, F. R. S. (Vol. XL., p. 330).

Author used tubes 10 c. m. long, closed with glass plates; which tubes had their ends inserted a short distance into perforations in the armature of an electro-magnet, as proposed by De la Rive. Water and carbon disulphide were used as standards of comparison. The results obtained bear little or no relationship to the chemical composition of the substance examined. But by referring the observations to lengths of tubes related to each other in proportion to their molecular weights, and making the necessary corrections for density, it is found that the molecular magnetic rotary power of bodies follows more or less regularly with the chemical composition.

A Spectroscopic Study of Chlorophyll. By W. J. RUSSELL, Ph. D., F. R. S., and W. LAPRAIK, F. C. S. (Vol. XL., p. 334).

By making an alcohol-ether solution of leaves, the authors obtained a chlorophyll solution and studied the four least refrangible absorption bands. Authors tried the action of hydrochloric acid gas upon the spectra and observed some remarkable movements in the position of the bands. They also state that other acids produce

like changes. Carbonic acid produces no change. Application of heat produces like changes. The action of mercuric chloride, ferric chloride is also noticed. Also the alum and basic lead precipitates. By adding alkalies to the chlorophyll solution, there is a fading out of all except the least refrangible band, and the spread of this band towards the blue. Further addition of alkali divides this band into two, and still more alkali entirely eliminates all but one band.

On the Precipitation of the Alums by Sodic Carbonate. By EDMUND J. WILLS, D. Sc., F. R. S., and K. L. BARR, (Vol. XL., p. 343).

By a series of experiments the authors find that the precipitation of an alum by sodic carbonate takes place in three stages. First, much carbonate is added without any precipitation taking place; secondly, there is a precipitation according to a continuous law until about half the alum is thrown down; and thirdly, the precipitation proceeds according to the previous law, but with altered constants.

On the Determination of Nitric Acid as Nitric Oxide by Means of its Reaction with Ferrous Salts. (Part II.) By ROBERT WARRINGTON. (Vov. XL., p. 345).

This paper gives the details of a modification of Schloesing's method with ferrrous chloride and hydrochloric acid, making the determinations in an atmosphere of carbon dioxide, measuring the nitric oxide by treatment with excess of oxygen in a gas analysis. It is especially applicable to the determination of nitrates in soils.

On the Determination of Nitric Acid in Soils. By ROBERT WARRINGTON. (Vol. XL., p. 351).

Author gives details of the manner of taking soil samples, the treatment of the samples, variations in the quantity of nitrates present in the soil resulting from different modes of drying, preparation of the watery extract, and analysis of the soil extract. Author also gives a set of tables showing the relative value of the Crum-Frankland and Schloesing methods for the determination of nitrates, giving the preference to the latter. The indigo method for the determination of nitric acid is shown to be valueless.

Metallic Compounds containing Bivalent Hydcocarbon Radicals. (Part III). By J. SAKURAI, F. C. S., University of Tokio, Japan. (Vol XL., p. 360).

By mixing monomercuric methylene iodide with mercuric chloride,

molecule for molecule, and cohabiting with alcohol, a white crystalline body was obtained, soluble in ether, chloroform, and alcohol, but insoluble in water. It melts at 129°C. Heated with a solution of iodine in potassic iodide, it yields an oily liquid. Its formula is given as CH_2 HgICl or monomercuric-methylene chloride. The constitution of this body is discussed and the formula Cl (CH_2) HgI is believed to be the correct one. Incidentally, during this investigation methylene chloroidide was obtained, and its boiling point is given as 109°C.

Some Observations on the Luminous Incomplete Combustion of Ether and other Organic Bodies. By W. H. Perkin, F.R.S. (Vol. XL., p. 363.)

The author noticed during the evaporation of ether that a pale blue flame, visible in the dark, was floating about upon the hot sand-bath near without igniting the ether which was evaporating. The temperature at which this phenomena occurs is about 260°C., from that to a dull red heat. A number of methods of exhibiting this phenomena at lectures are mentioned; one with a hot copper or iron ball, and another with a glass tube, which appear easy of execution. The temperature of this flame is low and will not ignite carbon disulphide. Another point about this combustion is that the quantity of carbon dioxide produced is very small. It was found that for 1.313 grm. of oxygen consumed only 0.133 grm. of carbon dioxide was found. Among other bodies that produce this effect are mentioned acetic aldehyde, the alcohols up to amylic pentane, hexane, heptane, and the hydrides up to paraffin. Benzol and homologues, phenol and cresol, give no result. The fatty acids give luminous effects; stearic acid at about 250°C. and when 290°C. is reached blue flames appear: oleic acid acts like stearic. Benzoic, cinnamic and phthalic acids give no results. Olive and linseed oils, and white wax act like oleic acid. Spermaceti shows the effect very well and is recommended for a lecture experiment. When solid paraffin was used and heated in a retort through which air was drawn 0.3816 grm. of oxygen was consumed and only 0.025 grm. of carbon dioxide obtained. Author thinks that the luminous appearance is similar to the phosphorescence obtained during the imperfect combustion of phosphorous. The light was too feeble for detailed spectroscopic analysis; but no bands were seen.

On Some New Compounds of Hæmatein and Brazilein. By J. J. Hummel and A. G. Perkin. From the Dye-house of the Yorkshire College. (Vol. XL., p. 367.)

Authors describe a simple method of preparing crystalline hæmatein in tolerable quantity. They use the ammonio-compound of hæmatein, and by treatment with strong acetic acid obtain crystals. These crystals are microscopic rhombic plates, soluble in water, alcohol, ether and acetic acid; and alkalies dissolve them readily. Analysis gave figures agreeing with the formula $C_{16}H_{12}O_6$ and the crystals are anhydrous.

By treating hæmatein with cold concentrated sulphuric acid and then adding acetic acid an orange colored crystalline powder is obtained, consisting of microscopic prisms; and analysis gives the formula $C_{16}H_{11}O_5HSO_4$ or *acid isohæmatein sulphate.* It is insoluble in alcohol, ether, and benzol; but is soluble to a small extent in acetic acid. Ammonia and soda solutions dissolve it.

When hæmatin is heated in sealed tubes with hydrochloric acid,and purified by crystallization from water microscopic orange-red needles are obtained, which analysis shows to have the formula $C_6H_{11}O_5Cl$. *Isohæmatein chlorhydrin.* It dissolves easily in water; it is less soluble in alcohol; and gives a reddish violet solution with alcoholic potash. Concentrated sulphuric acid converts it into isohæmatein sulphate.

Treatment of hæmatein with hydrobromic acid in the same manner as with hydrochloric acid gives *isohæmatein monobromhydrin* as microscopic needles, having the formula $C_{16}H_{11}O_5Br$ and resembling the chlorine compounds.

By treating isohæmatein chlorhydrin or bromhydrin with argentic hydrate, *isohæmatein* is obtained, having the formula $C_{16}H_{12}O_6$ and isomeric with hæmatein.

Crystalline Brazilein is obtained from Brazil Wood extract in the same manner that hæmatein is obtained from Logwood extract. It forms thin microscopic reddish-brown rhombic plates, very slightly soluble in cold water, more so in hot. Alkalies dissolve it. Analysis gives the formula $C_{16}H_{12}O_5H_2O$. Treated with concentrated sulphuric acid yellow rhombic needles of *acid isobrazilein sulphate* are obtained, having the formula $C_{16}H_{11}O_4HSO_4+H_2O$. It is slightly soluble in boiling glacial acetic acid, very soluble in alkalies.

By heating brazilein in sealed tubes with hydrochloric acid *isobrazilein chlorhydrin* as microscopic prisms is obtained, having the

formula $C_{16}H_{11}O_4Cl$. It is soluble in water and alkalies. By similar treatment with hydrobromic acid the corresponding bromine compound $C_{16}H_{11}O_4Br$. is obtained.

The tinctorial power of the new compounds is much greater than the original hæmatein and brazilein, and much faster.

On the Crystallization from Saturated Solutions of Certain Compound Salts. By JOHN M. THOMSON AND W. POFFLEWELL BLOXAM. (Vol. XL p. 379).

The Paper is a continuation of the experiments published in the *Journal* May 1879, on "The Action of Isomorphous Salts in producing the Crystallization of Supersaturated Saline Solutions." In the present communication the Authors take up the case of supersaturated solutions of double salts, acting upon them with nuclei consisting of one or the other of the component salts. Of the salts experimented with are the halogen salts of mercury with potassium and ammonium, potassic alum, Lefort's salt (the double zinc-copper sulphate), microcosmic salt, double tartrates and citrates of potassium, sodium and magnesium.

Abstracts from Berichte der Deutschen Chemischen Gesellschaft. By Percy Neymann, Ph.B.

On the Behavior of Chlorosulphurous Acid with Several Metalloids and with Tin. K. HEUMANN and P. KŒCHLIN. (Vol. XV. p. 416).

Chlorosulphurous acid $SO_2OH.Cl$. can be regarded as an acid and also as an acid chloride. The authors experimented with chlorosulphurous acid and sulphur, phosphorous, arsenic, antimony, tin, and carbon separately.

Chlorosulphurous acid and sulphur react upon heating and a yellowish oil, found by analysis to be sulphurous chloride S_2Cl_2, was distilled. Sulphurous acid gas and hydrochloric acid escaped during the reaction.

Chlorosulphurous acid and yellow phosphorus reacted best upon each other at a temperature of 25°—30° and currents of sulphurous and hydrochloric acid gases escaped. The liquid became more heated by the reaction and the experiment terminated in an explosion of the apparatus. Red phosphorous is only attacked after stronger heating and a few drops of a distillate are obtained which

seem to be phosphoroxychlorid, but this is probably the product of a second reaction.

Arsenic and antimony form their trichlorides. The reaction is characterized by leaving in both cases a non-volatile compound in the retort. The chlorosulphurous acid is most probably split up into chloride, sulphur dioxide and sulphuric acid: $2Cl.SO_3H = 2Cl + SO_2 + H_2SO_4$. The reaction with tin is much more complete the tetrachloride being formed according to the formula

$$\underset{Cl.}{4SO_2OH} + Sn = SnCl_4 + 2SO_2 + 2H_2SO_4$$

Freshly ignited wood charcoal when strongly heated with chlorosulphurous acid generates gases which cannot be liquefied. The decomposition of chlorosulphurous acid takes place as follows:

$$SO_2HCl + C = SO_2 + HCl + Cl.$$
$$\text{And } 2SO_2HCl + C = 2SO_2 + 2HCl + CO_2.$$

Chlorosulphurous acid hence has a direct chloridizing action upon sulphur, phosphorous, arsenic, antimony and tin, at the same time sulphurous anhydride and sulphuric acid are produced. The latter produces secondary reactions with the respective elements and their chlorides and hence oxygen compounds of the metalloids are formed. Carbon reacts in a different manner and produces $COCO_2HCl$ and SO_2.

Contributions to the Knowledge of Piperidine. C. SCHOTTEN (Vol. XV. p. 421).

Preparation of: 1. Amyl-piperidine from piperidine, amylbromide and aqueous solution of caustic potash.

2. Methyl-amyl-piperidine from amyl-piperidine and methyl iodide. Methyl-amyl-piperyl-ammonium-iodide is at first formed. The product of distillation of the ammonium base which was formed from the iodide by means of oxide of silver, almost completely dissolved in hydrochloric acid.

3. Benzyl-piperidine from benzyl-chloride and piperidine. After the reaction, water is added to the product and benzyl-piperidine is separated.

4. Methyl-benzyl-piperidine from benzyl-piperidine and methyl-iodide.

5. Nitroso-piperidine from piperidine and nitrous acid.

6. Piperyl-carbonicacidether by dropping ethyl-chloro-carbonate into piperidine. It is heavier than water and almost insoluble in it.

7. Acetyl and oxalyl-piperidine from acetyl-chloride and piperidine and oxalic ether and piperidine respectively. The author also produced pyridine from piperidine by means of bromine substitution products.

On the Dry Products of Distillation of Tartaric Acid. LEO LIEBERMANN. (Vol. XV. p. 428.). The products from 250 grms. of tartaric acid were:

Pyroracemic acid	9.24 grms.
Methylsuccinic acid	2.11 "
Formic acid	0.4 "
Tarry substances	3.0 "
Pitch-like substances, Aldehyde and Volatile acid	2.0 "

The larger proportion of the acid is hence decomposed according to this formula:

$$C_4H_6O_6 = C_3H_4O_3 + H_2O + CO_2.$$

A smaller portion thus:

$$2C_4H_6O_6 = C_5H_8O_4 + 2H_2O + 3CO_2.$$

and a third still smaller portion:

$$C_4H_6O_6 = CH_2O_2 + 2CH_2O + CO_2.$$

and judging from the copious generation of carbonic acid and carbonic oxide:

$$C_4H_6O_6 = H_2O + CO_2 + CO + 2CH_2O.$$

An Apparatus for Determining the Melting Point of Easily Liquified Metals and Alloys. LEO LIEBERMANN. (Vol. XV. p. 435). Description of apparatus.

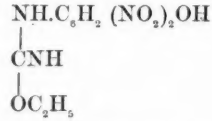
Sulphurous Acid in Wine. LEO LIEBERMANN. (Vol. XV. p. 437). Controversial.

Detection of Sulphurous Acid in Wine and other Liquids. LEO LIEBERMANN. (Vol. XV. p. 439). Two methods.

On the Optical Power of Rotation of Malic Acid and its Salts at Different Temperatures. Th. THOMSEN. (Vol. XV. p. 441). Results of experiments are tabulated.

The Action of Chlorine on Sulphonic Compounds and Organic Oxysulphides. W. SPRING and C. WINSSINGER. (Vol. XV. p. 445). Resumé of articles on the subject published in Bulletins de l'Académie de Belgique; III. Série. t. II. Déc. 1881.

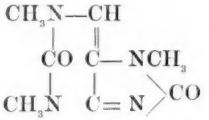
On the Action of Cyanogen upon Picraminic Acid. PETER GRIESS. (Vol. XV. p. 447). The author finds the formula for the product of a reaction between these two substances as



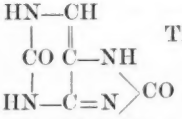
and terms it ethyl-oxy-carbimid-amido=dinitro-phenol.

On the Transformation of Xanthin into Theobromin and Coffein. EMIL FISCHER. (Vol. XV. p. 453). The author repeats an experiment, previously performed by *Strecker*, and by altering the conditions obtains evident proof of the constitution and formulæ, which latter are as follows:

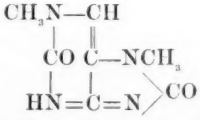
Coffein:



Xanthin



Theobromin :



On the Action of Iodine on the Silver Salts of some Acids of the Aromatic Series. K. BIRNBAUM and H. REINHERZ. (Vol. XV. p. 456). Experiments with benzoic, salicylic, and phtalic acids.

On the Critical Temperatures of Liquid Bodies. BR. PAWLEWSKI. (Vol. XV. p. 460). Controversial.

On Tropin. K. KRAUT. (Vol. XV. p. 462). Controversial.

Action of Pentacloride of Phosphorous on Acetyl and Benzoyl-diphenylamin. AL. CLAUS. (Vol. XV. p. 464). Controversial.

On same relations between Physical Constants of Chemical Compounds as set forth by Messrs. J. W. BRUEHL and V. ZENGER. (Vol. XV. p. 467).

On the Action of Bisulphide of Carbon on P-nitranilin. S. M. LOSANITSCH. (Vol. XV. p. 470). P-nitranilin and bisulphide of carbon boiled in alcoholic solution in the presence of caustic

potassa give rise to a thio-urethane, but not, as would be supposed, to a thio-urate. The analysis led to the formula

$$SO\begin{cases} NHC_6H_4NO_2 \\ OC_2H_5 \end{cases}$$

The compound is termed p-nitro-phenyl-xanthogenamid.

On the Action of Nitric Acid on Tribromanilin (common). S. M. Losanitsch. (Vol. XV. p. 471). Concentrated nitric acts energetically upon tribromanilin. The reaction is very complicated. 100 grms. of tribromanilin gave:

30 grms. dibrom-dinitro-methane.
5 " tetrabrombenzol.
10 " bromanil.
24 " dark red mass.

The remaining tribromanilin is converted into picric, oxalic and carbonic acids.

The action of nitric acid on tribromanilin in glacial acetic acid solution gives rise to the same products when boiled. If allowed only to be heated until red fumes appear dibrom-nitranilin is formed.

The action of nitric acid on tribromanilin in glacial acetic acid solution and alcohol results in the production of tribrombenzol.

On the Bases resulting from the Products of Addition of Chinalin and Halogen Alhydes. Ad. Claus. (Vol. XV. p. 475). Controversial.

On the Action of Amines on Chinones. Th. Zincke. (Vol. XV. p. 481). Continued from Vol. XIV.

On the Action of Amines on Dichlor Naphtochinone. A. Plagemann. (Vol. XV. p. 484). All experiments show that only one chlorine atom can be replaced by an amine rest, and that the second chlorine atom is not capable of any such interchange. The reaction takes place in accordance with the following equation: $C_{10}H_4Cl_2O_2 + NH_2R^1 = C_{10}H_4Cl.(NHR^1)O_2 + HCl$. The action of primary amines proceeds smoothly, and the resulting products can be classed in general with those of alpha naphtochinan. The action of secondary amines has also been experimented upon, and they have been found to act in the same manner.

On the Behavior of Nitrogen Tetroxide with Sulphuric Acid, and on the Process of Lasne and Benker for the Manufacture of Sulphuric Acid. G. Lunge. (Vol. XV. p. 488). In opposition to some previous statements, the author has

found that the tetroxide of nitrogen is not soluble as such in sulphuric acid, but is split up directly into nitrosyl sulphuric acid and nitric acid. $N_2O_4 + SO_2(OH)_2 = SO_2(OH)(ONO) + NO_3H$. The author proceeds to show that the theoretical assumptions of Lasne and Benker, given by these gentlemen in Vol. 92, p. 191, of Comptes Rendus, are not correct. This, however, does not mean that their process can not be of practical value.

On the Existence of Nitrous Anhydride in the form of Vapor. G. LUNGE. (Vol. XV. p. 495). Controversial.

On the Constitution of Cumic Acid. RICHARD MEYER and ERWIN MUELLER. (Vol XV. p. 496). The authors prepared cumic acid synthetically, and found a different melting point from that obtained in the usual manner, but have not yet been able to give any pronounced reasons. More extensive experiments are in progress, and results may be looked for soon.

On Tungsten Bronzes. J. PHILLIP. (Vol. XV. p. 499). Elaborate discussion on the preparation and analysis of various bronzes. There are four distinct colors, gold yellow, reddish yellow, red and blue. The yellow bronzes are most easily prepared pure, and can be obtained in large crystals. A mixture of 2 molecules of normal sodium tungstate, and 1 molecule of tungstic anhydride is employed (hence a mixture of 2 molecules of Na_2O to 3 molecules of WO_3). The tin is gradually added. The entire mixture is kept in quiet fusion for one or two hours. Red bronzes are obtained when 2 molecules of Na_2O and 5 or 6 molecules of WO_3 are melted with tin. Blue bronzes are obtained when more than 3 molecules of WO_3 to 1 molecule of Na_2O and tin are so treated. Tungsten bronzes are also prepared by reduction of acid sodium tungstates by means of hydrogen.

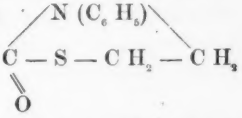
The Azoanthrol Dyes. C. LIEBERMANN. (Vol. XV. p. 510). The author prepared and tested a number of the dyes, and found that some of them dyed silk and wool directly, the tints produced being similar to the napthol dyes, falling between blood red and red brown. The dyes were prepared in the usual manner, by bringing solutions of diazatised bases or their sulpho acids, in contact with alkaline solutions of anthrol.

Contributions to the Thermometric Knowledge of Ozone. E. MULDER and H. G. L. VAN DER MEULEN. (Vol. XV., p. 511). Berthelot found the value for As_2O_3 aq 3000 as 137600^c, and the

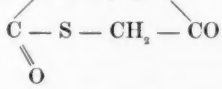
authors find that they can assume with appropriate certainty the figure 145000^c for $As_2 O_3$ aq 2000.

On Cyanic and Cyanuric Acid Ethers. J. PONOMAREFF. (Vol. XV. p. 513). The object of the experimentor is to study more closely the isomerism of the neutral and acid cyanuric acid ethers which are deduced from the salts of cyanuric acid, and which are formed by the action of bromcyanogen upon sodium ethylate. Experiments are still in progress.

On Mustard Oil Glycolide. PAUL T. MEYER. (Vol. XV. p. 516). It having been made possible to replace oxygen in the hydro-sulpho-diphenyl-hydantoïn of the group N ($C_6 H_5$) and to obtain a compound having the constitution



the author definitely assumes the constitution of phenyl mustard oil glycolide as



Action of Chlor Acetic Acid Ether on Phenylendiamin. J. ZIMMERMANN. (Vol. XV. p. 518). The product of a reaction between monochloracetic acid ethyl ether and phenylendiamin, 2 molecules of each, is a compound to which the author gives the formula

$$\begin{matrix} CH_2 - NH - COOC_2H_5 \\ > C_6H_4 \\ CH_2 - NH - COOC_2H_5. \end{matrix}$$

Continuation of Observations on the Behavior and Occurrence of Cinchotin, Hydrocinchonidin and Hydrochinidin. C. FORST and CHR. BOEHRINGER. (Vol. XV. p. 519). The result of experiments by the authors is that the basis obtained by oxidation were present in the original materials, and because of their great resistance remained unaltered.—*Ann. Chem. Pharm.*, 197–368.

On the Evolution of Free Nitrogen in Fermentation. B. E. DIETZELL. (Vol. XV., p. 551.) A series of experiments showed that nitrogen had been evolved in organic form as follows: I., 5.04 per cent.; II, 17.07 per cent.; III., 9.90 per cent.; IV., 8.97 per cent. The gases evolved from the fermentation of a mixture of blood and cow

urine were passed through dilute sulphuric acid, then over hot soda lime and titrated sulphuric acid. In all cases the sulphuric acid employed at the end remained unaltered. The author also found primary amines, leucin and free sulphurous acid. In presence of carbonate of lime solution no formation of ammonium nitrate can take place, and fatty acids which decompose nitrites cannot be formed, It seems to be advantageous to allow fertilizers, such as dried blood, bone dust, fish guano, etc., to ferment, with addition of a quantity of lime, until all nitrous acid has disappeared before they are brought into the receptacles containing the liquid excrement of domestic animals.

On Resorcin Dyes. G. Damm and L. Schreiner. (Vol. XV. p. 555.) Numerous attempts to obtain a body homologous to phenol-phatalein proved unsuccessful. A black mass was formed from which succinic acid could readily be obtained. Fusing succinic anhydride with resorcin in the air-bath at 140° resulted in the production of a body very similar to fluorescein. With bromine a body is produced which dyes similar to rosine.

On Halogen Derivatives of Chinolins. W. La Coste. (Vol. XV., p. 557.) The author prepares these derivatives in a manner differing from the original of Skraup, 86 grms. of bromanilin and the corresponding quantity of glycerin, sulphuric acid and nitrobenzol are carefully heated. The product was distilled with steam after diluting with water. The residue contained 70 grms. of pure monobromchinolin. Dinitrochinolin and phenylchinolin were prepared by similar methods.

On Normal Dithiurethanes. A. Bernthsen and G. Friese. (Vol. XV., p. 563.) Partly controversial. The results of experiments agree satisfactorily with those of W. Will. Ber. d. d. chem. Ges. XV. 338.

Remarks on a Note of C. Binzard and H. Schulz: On the Chemical Theory of Effects of Arsenic J. Dogiel. (Vol. XV., p. 572.) Controversial.

Remarks on M. Foerster's Article on the Presence of Furfurol in Fermented Liquids. W. A. Jorissen. (Vol. XV., p. 572.) Controversial.

The Condensation of Acetone. A. Pinner. (Vol. XV., p. 576.) Continued from Vol. XIV., p. 576. Cyanide of potash was to act upon acetone which allowed had been saturated with hydro-

chloric acid gas and thus condensed. Three combinations which could be easily separated were obtained; an acid $C_7H_{12}O_3$ called mesitonic acid, an acid of the composition $C_{15}H_{13}NO_3 + H_2O$ called (for the present) mesitylic acid, and a cyanide $C_{11}H_{18}N_2O_2$ from which an acid $C_{11}H_{18}O_5$ (phoronic acid) could be obtained.

Formation of Alloys by Pressure. W. SPRING. (Vol. XV., p. 595.) The author propounds the law, that the state of matter is dependent upon the volume it is forced to occupy. Coarsely powdered bismuth, cadmium and tin, mixed in the proportions corresponding to Wood's alloy, were subjected to a pressure of 7,500 atmospheres. This alloy melts at 65°. The block obtained was filed and again subjected to the same pressure. Its properties correspond exactly with those of Wood's alloy. Two other alloys, one known as Rose's and common brass, were treated in a similar manner, and the results obtained gave sufficient strength for the argument of the law set forth.

On Orthodinitro Compounds. AUG. LAUBENHEIMER. (Vol XV., p. 597.) Continued from Vols. IX., 768 and 1826; XI., 1155; XI., 303; IX., 760. The author finds that orthodinitrocompounds also react with sulphite of sodium.

On Bromchloral, Chlorobromal, Bromochloroform and Chlorobromoform. OSCAR JACOBSEN and R. NEUMEISTER. (Vol. XV., p. 599.). Description of these compounds obtained by the action of bromine on dichloracetal and monochloracetal and further decomposition of the products produced.

Bromdichloracetic Acid and Chlordibromacetic Acid. R. NEUMEISTER. (Vol. XV., p. 602.) When bromchloral or chlorobromal is heated with double its quantity of fuming nitric acid for about one hour, with vertical condenser, the corresponding substituted acetic acid crystallizes from it after cooling. Bromdichloracetic acid is $CCl_2Br.CO_2H$ and chlordibromacetic acid is $CClBr_2.CO_2H$. A number of derivatives of both compounds are described.

Note on Bodies Combustible with Difficulty. W. DEML (Vol. XV. p. 604.)

In elementary analysis some substances are burned imcompletely by the usual methods. The author finds an improvement in covering tbe substance to be analyzed in the platinum boat with three or four times its weight of previously ignited spongy platinum or platinum black.

On Chloromalonic Acid and its Derivatives. M. CONRAD and M. GUTHZEIT. Vol. XV. p. 605.

The free acid was produced from monochloromalonic acid ether by means of the potash salt. When monochloromalonic acid is heated until all carbon acid is driven off monochloracetic acid remains. By the action of ammonia upon monochloromalonic acid imido-dimalonyl-amid and amido-malonyl-amid were produced.

On Propylic and Isopropyl Succinic Acid. G. WALTZ. (Vol. XV. p. 608.)

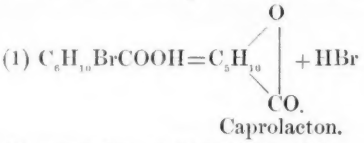
Description of method of preparation of derivatives.

On the Transformation of Alpha and Betanaphtal into Amidated Naphtalines. ARTHUR CALM. (Vol. XV. p. 609.)

Beta naphtylamine was obtained by the action of acetamid (ammoniumacetate) after distillation. The residue was found to be in part beta-acet-naphtalide, and in part beta-dinaphtylamine. A table giving the various temperatures and time required and results is appended.

On Caprolactone. ED. HJELT. (Vol. XV. p. 617.)

The caprolactone was obtained from monobrom-capranic acid with water The reactions take place as follows:



(1) $C_6H_{10}BrCOOH = C_5H_{10}\begin{matrix}O\\|\\CO\end{matrix} + HBr$

Caprolacton.

(2) $C_5H_{10}BrCOCH = C_5H_9COOH + HBr.$

Isohydrosorbinic acid.

Action of Water on Isodibromcapronic Acid. ED. HJELT. (Vol. XV. p. 619.)

A mixture of a lactone containing bromine and of a lactone free from bromine were the result of boiling dibromcapronic acid for one hour with ten times its weight of water. All bromocapronic acids prepared from sorbinic and hydrosorbinic acids, when heated with water or alkaline solution produce lactones.

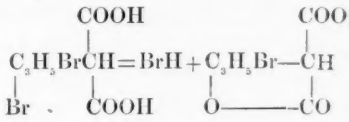
Oxypropylmalonic Acid and its Lactone. ED. HJELT. (Vol. XV. p. 621.)

Allylmalonic acid dissolves quite readily in fuming hydrobromic acid. The acid formed was found to be the lactonic acid, having

the same composition as the allylmalonic acid. It is a monobasic lactonic acid, the lactone of an oxypropylmalonic acid.

Action of Bromine on Allylmalonic Acid. Ed. Hjelt. (Vol. XV. p. 624.)

Besides the dibromide an oil poorer in bromine is produced. The reaction takes place as follows:



On Dilactones. Ed. Hjelt. (Vol. XV. p. 625.)

By the action of 2 mol. of bromine upon diallylmalonic acid a dibromide is produced. Hydrobromic acid is freely produced. The quantity of bromine present as hydrobromic acid after the reaction is completed was 50 per cent. of the amount of bromine employed. The bromide being a neutral body its formation can only be comprehended by assuming the formation of a tetrabromide at first, from which 2 mol. of bromine were driven which combined with the two hydrogen atoms of the carboxyls. The compound is hence an example of a dilactone.

Action of Hydrobromic Acid and Bromine on Diallylacetic Acid. Ed. Hjelt. (Vol. XV. p. 627.)

Hydrobromic acid reacts with diallylacetic acid forming a thick oil termed brom-dipropyl-aceto-lactone. Bromine reacts with it, producing tribrom-dipropyl-aceto-lactone.

On the Boiling Points of Lactones. Ed. Hjelt. (Vol. XV. p. 629.)

Gives the boiling points of lactones hitherto discovered.

On the Formation of Lactone. Ed. Hjelt. (Vol. XV. p. 629.)

From the researches of Fittig it is comparatively certain that the lactones are generated from the oxyacids and bromine substitution acids, which have their hydroxyl group or the bromine atom respectively attached to the third carbon atom, beginning at the carboxyl group. For this theory the investigations of the author on the addition products of bromine and hydrobromic acid with allylmalonic, diallylmalonic and diallylacetic acids give sufficient evidence.

Abstracts from the Comptes Rendus; by A. Bourgougnon.

Action of Ethylenic Chlorhydrine upon the Pyridc bases and Quinolein. By A Wurtz. (No. 6, Aug. 7th, 1882).

Upon the Composition of the Iodides of Phosphorus. By L. Froost. (No. 6. Aug. 7th, 1882).

The composition off the Iodides of Phosphorus is represented by PI_3 and P_2I_4.

Upon Quinolein and Lutidin. By A. Pictet. (No. 6. Aug. 7th, 1882).

Neutral Arsenates. By E. Filhol & Senderns. (No. 7. Aug. 14th, 1882).

The arsenates $2\ As_2O_5\ 3\ Na.\ OH + 6\ H_2O$ and $+4\ As_2O_5\ 3\ Na\ OH\ 3\ KOH + 18\ H_2O$ have no action upon litmus.

Upon the the Chemical Composition of the ripe and unripe Banana. By L. Ricciardi. (No. 8. Aug. 21st, 1882).

	Ripe.	Unripe.
Water	66.78	7.92
Cellulose	0.17	0.36
Starch	traces	12.06
Tannic substances	0.34	6.53
Fatty "	0.58	0.21
Interverted sugar	20.07	0.08
Cane sugar	4.50	1.34
Proteic substances	4.92	3.04
Not determined	1.69	4.42
Ashes	0.95	1.04

The green banana contains a large amount of starch, about $\frac{1}{8}$ of its weight which disappears in the ripe fruit. When the fruits are left to ripen on the trees nearly all the sugar formed is cane sugar, whilst the sugar contained in the fruits taken from the trees in a green state and allowed to ripen afterwards is composed of $\frac{4}{5}$ of interverted sugar and $\frac{1}{5}$ of cane sugar. The tannic substances and the organic acids of the green fruits disappears when the bananas are ripe.

The ashes of bananas are very rich in phosphoric acid (23.18 per cent.) and potassium oxide (45.23 per cent.)

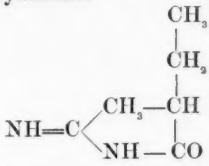
On Black Phosphorus. By P. Thenard. (No. 9. Aug. 28th, 1882).

Separation of Gallium. By L. DE BOISBAUDRAN. (No. 9. Aug. 28th, 1882).

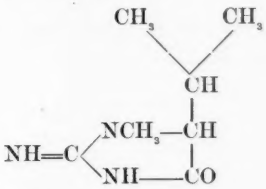
Separation of Gallium from Indium and Cadmium.

On some combinations belonging to creatin groups. By E. DUVILLIER. (No. 10, Sept. 4th, 1882).

The following combinations have been obtained: 1° Methylamido Alpha-butyrocyamidin:



2° Methylamids isovalerocyamidin.



Separation of Gallium. By L. de BOISBAUDRAN. (No. 12 Sept. 18th, 1882).

Separation of Gallium from Uranium and Lead.

LIST OF

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MEMBERS AND ASSOCIATES

—OF THE—

AMERICAN CHEMICAL SOCIETY.

DECEMBER, 1882.

NEW YORK:
BERMINGHAM & COMPANY, PUBLISHERS.
28 UNION SQUARE,
1882.

OFFICERS

—OF THE—

AMERICAN CHEMICAL SOCIETY,

For the Year 1882.

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BUNSEN, R. W., University of Heidelberg, Germany.

CANNIZZARO, STANISLAS, Universita Instituto Chemico della Regia, Rome, Italy.

FRANKLAND, E., Royal College of Chemistry, South Kensington Museum, London, England.

WILLIAMSON, A. W., University College, London, W. C., England.

WOEHLER, Fr.*

MEMBERS.

LIFE MEMBERS ARE MARKED ‖.
DECEASED MEMBERS ARE MARKED *.

DATE OF ELECTION.		
April	6, 1876	Adams, Isaac, Jr., 329 Broadway N. Y.
Dec.	7, 1876	Aitken, Andrew P., Clyde S., Edinburgh, Scotland.
April	6, 1876	Alsberg, M., 54 Maiden Lane, N. Y.
April	6, 1876	Amend, B. G., 205 Third ave., N. Y.
May	3, 1877	Appleton, J. H., Brown University, Providence, R. I.
April	6, 1876	Atwater, Wilbur O., Middletown, Conn.
Dec.	7, 1876	Austen, P. T., Rutger's College, New Brunswick N. J.
July	6, 1876	Bailey, E. H., Bethlehem, Pa.
March	7, 1878	Baker, W. H., Freeport, Ill.
April	6, 1876	Balentine, Walter, Argicultural Department, Washington, D. C.
Oct.	5, 1876	Barcena, Mariano, Museo Nacional, City of Mexico, Mexico.
April	6, 1876	Battershall, J. P., 76 Varick st., N. Y.
Oct.	7, 1881	Bauer, Harry von, 12 Frankford st., N. Y.
June	1, 1876	Baumgarten, A., St. Lawrence Sugar Refining Co., Montreal, Canada.
April	6, 1876	Baumgarten, F. M., 179 16th st., San Francisco, Cal.
Jan.	3, 1878	Beckwith, Leonard F., Courtlandt and Church sts., N. Y.
April	6, 1876	Behr, Arno, care of Matthiessen & Wiechers, Jersey City N. J.
July	6, 1876	Belcher, G. W., St. Louis, Mo.
April	3, 1879	Benjamin, Marcus, 6 Barclay st., N. Y.
Oct.	7, 1880	Bihn, G. T., 523 Tasker st., Philadelphia, Pa.
Nov.	1, 1877	Blanpied, Benj., Dartmouth Agricultural Coll., Hanover, N.H.
April	6, 1876	*Blossom, Thomas M.
Dec.	7, 1876	Booth, J. C., United States Mint. Philadelphia, Pa.
April	6, 1876	Bourgougnon, A., 26 W. 4th St., N. Y.
March	1, 1880	Bragg, Everett B., 33 Park place, N. Y.
Jan.	6 1882	Braschi, Victor M., School of Mines, N. Y.
April	7, 1882	Brenneman, A. A., Cornell Univ'y, Ithaca N. Y.
April	6, 1876	Brinley, Charles A., Midvale Steel Works, Germantown, Philadelphia, Pa.
Dec.	6, 1877	Brown, Wm. G., East Tennessee University, Knoxville, Tenn., Box 596.
Sept.	7, 1876	Buck, C. Elton, Wilmington, Del.

DATE OF ELECTION.

April 6, 1876 *Cairns, F. A.,
May 2, 1878 Calder, E. E., Box 17, Providence, R I.
April 6, 1876 Casamajor, P., care Havemeyer & Elder, Brooklyn, E. D., N. Y.
April 6, 1876 ‖ Chandler, Chas. F., School of Mines, Columbia College, N. Y.
Sept. 7, 1877 Chase, Alonzo, Room 123, Cochrane B'ld'g, Washington, D.C.
April 6, 1876 Chandler, W. H., Bethlehem, Pa.
April 6, 1876 Chester, Albert H., Clinton, N. Y.
Oct. 7, 1881 Colby, Albert L., School of Mines, N. Y.
March 7, 1878 Colby, E. Edwards, School of Mines, Columbia College N. Y.
Dec. 6, 1879 Convers, G. G., U. S. Mint. New Orleans, La.,
Feb. 1, 1877 Corning, A. J., N. E. cor. Bolton & Mosher sts., Baltimore, Md
Nov. 7, 1878 Cruse, E. F., 20 Bridge st., Brooklyn, N. Y.

Dec. 6, 1877 Dabney, Charles W., Raleigh, N. C.
Nov. 4, 1881 Darton, Nelson H., 119 Pearl st., N. Y.
Sept. 5, 1878 Debrunner, Henry G., Iron City, via Alpine Chaffee Co., Co.
April 6, 1876 Degenhardt, F. A. C., 333 S. 5th st., Brooklyn, N. Y.
April 6, 1876 Doremus, Charles A., 92 Lexington ave. N. Y.
Dec. 2, 1879 Dosher, Henry, 142 Rose st., Brooklyn, E. D.
April 6, 1876 Douglass, Silas H., Ann Arbor, Mich.
April 6, 1876 * Draper, John W.
Nov. 2, 1876 Dudley, Charles G., Altoona, Pa
July 6, 1876 Dunnington, F P., University of Virginia, Albemarle Co., Va.
May 3, 1877 Dwight, Charles Edgar, Wheeling, W. Va.

April 6, 1876 Eastwick, E. P., 78 Wall st., N. Y.
April 6, 1876 Ebert, A. E., Peoria Sugar Refinery, Peoria, Ill.
March 6, 1879 Eimer, August, 220 E. 17th st., N. Y.
April 6, 1876 Elliott, Arthur H., School of Mines, Columbia College, N. Y.
Oct. 5, 1876 Ellis, William H., Toronto, Canada.
April 6, 1876 Endemann, H., 33 Nassau st., N. Y.
April 6, 1876 Englehardt, Francis E., Syracuse Salt Co., Syracuse, N. Y.
March 6, 1879 Erhardt, C. F., care Chas. Pfizer & Co., 81 Maiden Lane N. Y.

April 6, 1876 Fesquet, Adolphe A., 1322 Marshall st., Philadelphia, Pa.
June 1, 1876 Fisher, R. A., 2239 St. Albans st., Philadelphia, Pa.
Dec. 2, 1879 Freish, H., 136 Church st., Bridenburg, Philadelphia, Pa.
Dec. 2, 1880 Friedburg, L. H., 40 E. 10 st., N. Y.
April 6, 1876 Fristoe, Edward E., Columbian College, Washington, D. C.

May 1, 1879 Gallatin, Albert H., Room 26, Cooper Union, N. Y.
April 6, 1876 Genth, Fred. A., University of Pennsylvania, Phila., Pa.
Feb. 11, 1881 Gerber, N., Amer. Cond. Milk Co., Little Falls, N. Y.
April 6, 1876 Gesner, G. W., 81 John st. N. Y.
March 1, 1877 Geyer, Wm. E., Stevens Institute, Hoboken, N. J.
Oct. 14, 1879 Giessecke, Dr., 19 W. Swan st., Buffalo, N. Y.
April 2, 1882 Gladding, Thomas S., 55 Fulton st., N. Y.
April 6, 1876 Goessman, Charles A., Amherst, Mass.
April 6, 1876 * Goldmark, J.
Oct. 5, 1876 Goldschmidt, S. A., 55 Water St., Brooklyn.

DATE OF ELECTION.	
April 6, 1876	Green, Trail, Easton, Pa.
June 7, 1877	Gregg, W. H., Elmira, N. Y.
May 6, 1881	Grote, Otto, 59-61 Goerck st., Y. Y.
April 6, 1876	Grund, F. A., 106 Wall st., N. Y.
April 6, 1876	Habirshaw, William M., 159 Front st., N. Y.
Dec. 2, 1880	Hale, Albert C., State School of Mines, Golden, Col.
June 3, 1881	Hallock, A. P., N. Y. Gaslight Co., ave. A and 21st st., N. Y.
Oct. 7, 1881	Habel, Louis, 287 Leonard st., Brooklyn, N. Y.
Oct. 2, 1879	Hamer, Thomas H., Nassau Bank, N. Y.
April 6, 1876	Hardin, M. B., Virginia Military Institute, Lexington, Va.
Oct. 7, 1880	Harrison, Thomas, Philadelphia, Pa.
April 6, 1876	Hasenclever, Maurice, 1 Appleton st., Lawrence. Mass.
June 1, 1876	Havemeyer, Hector C., 89 Wall st., N. Y.
April 6, 1876	Hayes, S. Dana, 4 State st., Boston, Mass.
Feb. 6, 1879	Hazard C. care Caswell, Hazard & Co., 5th Ave. Hotel, N. Y.
April 6, 1876	Hedrick, Benjamin S., U. S. Patent Office, Washington, D. C.
June 1, 1876	Henes, W. F., 1620 Third ave., N. Y.
April 6, 1876	Herreshoff, J. B. F., 135 Clinton st., Brooklyn, N. Y.
Oct. 7, 1881	Hiepe, H. C., 102 John st., N. Y.
April 6, 1876	Hill, Nathaniel P., Denver, Col.
Jan. 4, 1877	Hill, W. M., U. S. Torpedo Station, Newport, R. I.
March 1, 1877	Hindley, Robert C., Racine College, Racine, Wis.
March 3, 1882	Hirshberg, Emil, 16 Dey st., N. Y.
April 6, 1876	Homer, Chas. S., care of Valentine & Co., 323 Pearl st., N. Y.
Feb. 11, 1881	Hopke, T. M., 17 Cedar st., N. Y.
April 1, 1881	Hoppock, A. E., 103 E. 148th st., N. Y.
Jan. 4, 1877	Horsford, E. N., Cambridge, Mass.
April 6, 1876	Humphrey, Henry C., Stamford, Ct.
April 6, 1876	Hunt, T. Sterry, 33 Durocher st., Montreal, Canada.
June 6, 1878	Jenks, A. M., care of Havemeyer, Eastwick & Co., Jersey City, N. J.
July 6, 1876	Johnson, Samuel W., New Haven, Ct.
March 6, 1879	Kelly, John F., 97 Garden st., Hoboken, N. J.
April 6, 1876	Kersting, Herman, 324 Princeton st., East Boston, Mass.
July 6, 1876	Koenig, G. A., University of Pennsylvania, Philadelphia, Pa.
April 6, 1876	Krackowitzer, S., 512 E. 17th st., N. Y.
April 6, 1876	Krause, O. H., care Matthiessen & Wiechers, Jersey City, N. J
April 7, 1882	Krause, G. Chemiker Zietung, Coethen, Germany.
Oct. 7, 1881	Lauver, Winfield P., care of Treasury Department, Washington, D. C.
July 6, 1876	Lawler, C. J., cor. Bedford and Gates aves., Brooklyn, N. Y.
Dec. 7, 1876	Lecour, E. H., 547 W. 18th st., N. Y.
April 6, 1876	Ledoux, A. R., 17 Cedar st., N. Y.
Jan. 3, 1878	Leeds, A. R., Stevens Institute, Hoboken, N. J.
Jan. 3, 1878	Lees, Wm. T., N. Y. Gaslight Co., ave. A and 31st st., N. Y.
April 6, 1876	Liebig, G. A., 87 Exchange place, Baltimore, Md.

DATE OF ELECTION.

July 6, 1876 Lillie, Samuel Morris, 333 Pine st., Philadelphia, Pa.
Oct. 7, 1881 Leman, W. P., 305 W. 55th st., N. Y.
April 6, 1876 Lindsley, J. Berrian, Nashville, Tenn.
April 6, 1876 *Lipps, John S.
Feb. 3, 1882 Lugo, Orazio, University, Burling, N. Y.
Dec. 2, 1880 Lungwitz, Theo., care of H. E. Niese, Jersey City, N. J.
April 6, 1876 Lupton, N. T., Vanderbilt University, Nashville, Tenn.
May 2, 1878 Luthy, D., 220 Church st., Philadelphia, Pa.

April 6, 1876 McIntire, Chas. M., Jr., 311 N. Hampton st., Easton, Pa.
Jan. 2, 1879 *McIntire, Henry M.
June 6, 1878 MacIntosh, J. B., 55 Garden st., Hoboken, N. J.
April 6, 1876 Maish, John M., College of Pharmacy, Philadelphia, Pa.
April 6, 1876 Mallet, J. W., University of Virginia, Albemarle Co., Va.
Feb. 6, 1879 Marchand, Charles, P. O. Box 1567, N. Y.
April 6, 1876 Martin, Wm. J., Davidson College, Mecklenburg Co., N. C.
Sept. 7, 1876 Martins, R. A., care of Actien-Gesellschaft fuer Anilin Fabrikation, Berlin, Germany.
May 6, 1878 Mattison, J. G., Hastings-on-Hudson, N. Y.
Feb. 1, 1877 Means, Alexander, Oxford, Newton Co., Ga.
April 6, 1876 *Merrick, John M.
April 6, 1876 Mew, Wm. M., 422 5th st., Washington, D. C.
Dec. 1, 1879 Meyers, Henry C., P. O. Box 3037 N. Y.
June 1, 1876 Miller, George M., 20 Broad st., N. Y.
April 6, 1876 Moore, Gideon E., 69 Liberty st., N. Y.
April 6, 1876 Morrill, Thomas T., Cambria Iron Works, Johnstown, Pa.
April 6, 1876 Morton, Henry, Stevens Institute, Hoboken, N. J.
June 1, 1871 Mott, H. A. Jr., 105 Water st., N. Y.
April 6, 1876 Munroe, Charles E., U. S. Naval Academy, Annapolis, Md.
March 7, 1878 Munroe, H. S., School of Mines, Columbia College, N. Y.
Nov. 4, 1881 Munsell, C. E., 128 Worth st., N. Y.

April 6, 1876 Nason, Henry B., Troy, N. Y.
April 6, 1876 Niese, H. E., care of Matthiessen & Wiechers, Jersey City, N. J.
April 6, 1876 Nichols, W. H., 41 Cedar st., N. Y.
May 3, 1877 Norton, Thomas, 2 Route Landai, Saint Denis, Paris, France.

Nov 4, 1881 O'Connor, Thos. D., 42 W. 28th st., N. Y.
Dec. 2, 1880 O'Connor, J. D., School of Mines, Columbia College, N. Y.
Nov. 19, 1879 Oothout, William, Jr., 218 Madison ave. N. Y.
Dec. 6, 1877 Orth, A., care of F. Bredt & Co., N. Y.

July 6, 1876 Parker, T. J., Box 69, Bergen Point, N. J.
April 6, 1876 Parsons, C. C., 542 E. 20th st., N. Y.
July 6, 1876 Pearce, Richard, Denver, Col.
May 3, 1877 Penissat, A., 41 Cedar st., N. Y.
April 7, 1882 Perkins, G. E., Providence, R. I.
April 6, 1876 Perkins, Maurice, Schenectady, N. Y

DATE OF ELECTION.	
Jan. 6, 1882	Peters, J. A., 92 William st., N. Y.
April 6, 1876	Phelps, George N., 127 First place, Brooklyn, N. Y.
July 11, 1878	Phillips, William B., University of North Carolina, Chapel Hill, N. C.
Nov. 2, 1876	Pickhardt, Carl W., Pickhardt & Kuttroff, cor. of Liberty and New Church sts., N. Y.
Dec. 2, 1880	Pigeon, Narcise, 421 W. 15th st., N. Y.
April 6, 1876	Pirz, Anthony, Long Island City, N. Y.
Oct. 5, 1876	Pitkin, Charles A., Box 52, Braintree, Mass.
Jan. 6, 1882	Pitkin, Lucius, 432 Madison ave., N. Y.
March 6, 1879	Pool, Franklin V., 191 Roseville ave., Newark, N. J.
Oct. 7, 1880	Poole, Herman, Buffalo, N. Y.
April 6, 1876	Prescott, Albert B., Ann Arbor, Mich.
March 1, 1877	Prochazka, George A., 162 2d ave., N. Y.
Oct. 7, 1881	Radenhausen, P., Stevens Institute, Hoboken, N. J.
Jan. 6, 1882	Rau, Henry, 92 Church st., N. Y.
April. 7, 1882	Remsen, Robert G., Jr., 26 Waverly place, N. Y.
April 6, 1876	Rice, Charles, Bellevue Hospital, N. Y.
April 6, 1876	Ricketts, P. de P., School of Mines, Columbia College, N. Y
April 6, 1876	Rising, William B., University of California, Oakland, Cal.
June 1, 1876	Robbins, Charles A., 91 Fulton st., N. Y.
March 4, 1878	Roberts, W. Chandler, Royal Mint, London, Eng.
March 4, 1878	Rogers, F. M., 95 Liberty st., N. Y.
April 6, 1876	Rogers, Robert E., Jefferson Medical College, Phila., Pa.
Nov. 1, 1877	Rossi, Auguste J., 22 W. 11th st., N. Y.
July 11, 1878	Rupp, William, 117 Pearl st., N. Y.
April 6, 1876	*St. John, Samuel.
June 5, 1879	Schanck, J. Stillwell, Princeton College, Princeton, N. J.
April 6, 1876	Schultz, Charles H., cor. 17th st. and Broadway, N. Y.
April 6, 1976	Schweitzer, Paul, Columbia, Boone Co., Mo.
Dec. 2, 1880	Seaman, Henry J., Crane Iron Co., Catasauqua, Pa.
July 6, 1876	Seeley, Henry Martyn, Middlebury, Vt.
Jan. 1, 1880	Semper, C., Gray's Ferry Chem. Works, Philadelphia, Pa
April 6, 1876	Shapleigh, Waldron, Freeport, Ill.
Nov. 4, 1881	Share, W. W., 336 Navy st., Brooklyn, N. Y.
April 6, 1876	Sharpless, S. P., 114 State st., Boston, Mass.
Oct. 3, 1878	Shepard, Charles U., jr., 20 Broad st., Charleston, S. C.
April 6, 1576	Sherer, E., 122 Front st., N. Y.
April 6, 1876	Silliman, Benjamin, New Haven, Ct.
April 6, 1876	Simon, Wm., Maryland College of Pharmacy, Baltimore, Md.
Feb. 11, 1881	Slade, James F., 5 E. 38th st., N. Y.
May 3, 1877	Sloane, T. O'C., 55 Pine st., N. Y.
Nov. 1, 1877	Smith, Hanbury, 35 Union square, N. Y.
March 1, 1877	Squibb, E. R., 36 Doughty st., Brooklyn, N. Y.
June 5, 1879	Stebbins, James H., Jr., 20 E. 37th st., N. Y.
April 6, 1876	Sticht, John C., care of Charles Pfizer & Co., 81 Maiden Lane, N. Y.

DATE OF ELECTION.	
April	6, 1876 Stillwell, Charles M . 55 Fulton st., N. Y.
July	6, 1876 Sweeny, William S., Easton, Pa.
April	6, 1876 Taylor, William H , 606 E. Grace st., Richmond, Va.
Oct.	5, 1876 Terne, Bruno, 1628 S. 5th st., Philadelphia, Pa.
Feb.	7, 1878 Thompson, A. L., 52 Sharp st., Baltimore, Md.
May	1, 1881 Tonnelé, Theodore, Wampum Cement and Lime Co., Newcastle, Lawrence Co., Pa.
Oct.	7, 1880 Torrey. C. H., School of Mines, N. Y.
Feb.	7, 1878 Tscherniac, J., 118 Boulevard Maillot, Neuilly, Paris, France.
May	6, 1879 Tucker, J. H., care of McKean, Newhall & Borie, Philadelphia, Pa.
April	3, 1879 Van Slooten William, 42 S. Derbigny st., New Orleans, La.
April	6, 1876 Vaughn, Henry W., Proviflence, R. I,
Feb.	11, 1881 Venable, F. P., Chapel Hill, N. C.
Nov.	4, 1881 Volney, C. W., Mott ave., N. Y.
Dec.	6, 1877 Wait, Charles E., Missouri School of Mines, Rolla, Mo.
Jan.	3, 1878 Waldstein, Martin E., 92 New Church st., N. Y.
April	6, 1876 Waller, Elwyn, School of Mines, Columbia College, N. Y.
April	6, 1876 *Walz, Isidor.
June	6, 1878 Ware, Louis S., St. George Hotel, Philadelphia, Pa.
April	6, 1876 Weber, Henry A., Ill. Industrial University, Champaigne, Ill.
April	6, 1876 Wendt, Charles J., care of Charles Pfizer & Co., 81 Maiden Lane, N. Y.
Sept.	7, 1876 Wenzell, Wm. T., 852 Market st., San Francisco, Cal.
March	6, 1879 Wheeler, C, G., University of Chicago, Chicago, Ill.
Dec.	2, 1880 Wiechman, F. G., School of Mines, N. Y.
Jan.	2, 1879 Wigner, G. W., 79 Great Tower st., London, England.
Dec.	1, 1879 Williams, E. H., 101 N. 32d st., Philadelphia, Pa.
June	5, 1879 Wilson, M. C., Waco, Texas.
April	6, 1876 Winans, Horatio N., 1551 Broadway, N. Y.
Nov.	4, 1881 Witthaus, R. A., Buffalo Med. College, Buffalo, N. Y.
Dec.	2, 1880 Wittman, Byron N., Pittsburg Steel Casting Co., Pittsburg, Pa.
July	6, 1876 Wolf, Theo. R., Delaware College, Newark, Del.
April	6, 1876 Wormley, Theo. G., University of Pennsylvania, Phila., Pa.
July	6, 1876 Youmans, W. J., care of D. Appleton & Co., 1 Bond st., N. Y.

Associate Members.

DATE OF ELECTION.		
June	1, 1876	Adler, J., 744 Lexington ave., N. Y.
June	1, 1876	Bartlett, Willard, 110 Broadway, N.Y.
March	6, 1879	Binns, Joseph, cor Pearl and Cedar sts., N. Y.
March	4, 1880	Booraem, J. V. V., N 3d and 1st sts., Brooklyn, E.D., N. Y.
June	6, 1878	Bower, Henry, Gray's Ferry Road, Philadelphia Pa.
June	3, 1880	Cochrane, A., 55 Kilby st., Boston, Mass.
June	3, 1880	Cochrane, Hugh, 55 Kilby st., Boston, Mass
July	6, 1876	Day, Austin G., 120 Broadway, N. Y.
June	1, 1876	Day, Walter De F., 301 Mott st., N. Y.
July	6, 1876	Dickerson, Edward N., Staats-Zeitung Building, N. Y.
April	5, 1877	Donner, J. O., care of Decastro & Donner, foot of South 9th st., Brooklyn, N. Y.
Feb.	11, 1881	Dunham, E. K., School of Mines, N. Y.
Jan.	4, 1877	Elder, Fred H., care of Havemeyer & Elder, 117 Wall st., N. Y.
July	6, 1876	Elder, George, Jr., 19 West 48th st., N. Y.
Oct.	5, 1876	Elmenhorst, W. R., St, Lawrence Sugar Refining Co., Montreal, Canada.
July	6, 1876	Gandolfo, Joseph, 41 Cedar st., N. Y.
April	4, 1878	Harding, G., 237 Broadway, N Y.
Sept.	7, 1876	Havemeyer, C. W., care of Havemeyer Bros., Brooklyn, E. D., N. Y.
April	5, 1877	Havemeyer, Henry, 89 Wall st., N. Y.
July	6, 1876	Havemeyer, Henry O., 117 Wall st., N. Y.
July	6, 1876	Havemeyer, Theodore A., 117 Wall st., N. Y.
April	5, 1877	Heller, Fred., 55 Maiden Lane, N. Y.
June	1, 1866	Hinds, Joseph, care of Havemeyer & Elder, Brookly, E. D., N. Y.
Dec.	6, 1876	Holly, John J., 63 Beaver st., N. Y.
June	1, 1876	Holt, Henry, 12 East 23d st., N. Y.
April	5, 1877	Kalbfleisch, A. M., cor. Fulton and Cliff sts., N. Y.
April	5, 1877	Kalbfleisch, Franklin A., cor. Fulton and Cliff sts., N. Y.
Nov.	7, 1876	Kuttroff, Adolph, Wm., Pickhardt & Kuttroff, cor. New Church and Liberty sts., N. Y.
Dec.	2, 1880	Kyte, Charles, Jr., 3 East India ave., London, E. C.

DATE OF ELECTION.		
Jan.	6, 1882	Le Boutillier, Clement, School of Mines, N. Y.
Feb.	6, 1879	Leggett, E. W., Elizabethport, N. J.
March	1, 1877	Lennig, Charles, Philadelphia, Pa.
July	6, 1876	Mapes, Charles V., 158 Front st., N. Y.
July	6, 1876	Matthiessen, F. O., 106 Wall st., N. Y.
April	5, 1877	Merz, Henry, care of Heller & Merz, 55 Maiden Lane, N. Y.
July	6, 1877	Miles, William A., 57 Christie st., N. Y.
April	5, 1877	Nichols, G. H., 41 Cedar st., N. Y.
Sept.	7, 1876	Page, George, Shepherd, 49 Wall st., N. Y.
July	6, 1876	Parsons, W. J., 66 Water st., Brooklyn, N. Y.
July	6, 1876	Plympton, G. M., 245 Broadway, N. Y.
Nov.	2, 1876 ‖	Pickhardt, William, Wm. Pickhardt & Kuttroff, cor. New Church and Liberty sts., N. Y.
March	7, 1878 ‖	Prentice, W. P., 301 Mott st., N. Y.
May	3, 1877	Renwick, Edward S., 16 Murray st., N. Y.
Sept.	7, 1876	*Russell, W D.
Jan.	6, 1882	Rudesch, J., 117 East 59th st.
May	6, 1881	Sawyer, C. P., High Point, Guilford Co., N. C.
Oct.	5, 1876 ‖	Senff, Charles H., care of Havemeyer & Elder, 117 Wall st., N. Y.
July	6, 1876	Senff, Frederick W., 558 Madison ave., N. Y.
April	5, 1877	Schroeder, E., 100 Glenwood ave., Jersey City, N. J.
April	5, 1877	Sieghoertner, A. L., Belcher Sugar Refining Co., St. Louis, Mo.
Jan.	4, 1877	Squier, Stewart C., 113 Liberty st., N. Y.
April	5, 1877	Stursberg, Julius A., Brooklyn Sugar Refinery, foot of South 9th st., Brooklyn, N. Y.
May	6, 1881	Van Sinderen, A. Howard, School of Mines, N. Y.
Dec.	2, 1880	Vulte, Herman T., School of Mines, N. Y.
May	2, 1878	Weston, E. D., 135 Lake st., Chicago, Ill.
May	2, 1878	Williamson, D. D., 683 Madison ave., N. Y.
Jan.	6, 1882	Wittmack, Chas. A., School of Mines, N. Y.
March	6, 1879	Ziegler, William, care of Royal Baking Powder Co., 91 Duane st., N. Y.

PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

Regular Meeting, Dec. 8, 1882.

The meeting was called to order at 8.40 P. M. Prof. A. R. Leeds in the chair.

The minutes of the previous meeting were read and approved.

The board of directors had no report. The treasurer stated that we had a balance of about $200 in the treasury.

The librarian and curator had no report. The committee on endowment fund, stated that the labor of raising the fund was for the present suspended, there being a larger fund in the possession of the society than was needed for the publication of the original papers presented at the present time.

The committee on papers and publications then stated that it had no formal report to present, but trusted that the Society would judge them by the work they had done.

The committee further stated that the November number of journal is in type, and that it will probably be issued the coming week.

The next in order was the election of officers for the ensuing year.

For President.—The three following gentlemen received the greatest number of votes: A. R. Leeds, E. R. Squibb, J. C. Booth.

Prof. Leeds declined to be a candidate, on the ground that it was of the greatest importance to the interests of the Society, as a national body, that the honorary office of President should be filled by a non-resident member.

It was then moved that the Society proceed to vote for the two other gentlemen standing highest on the list. This being seconded was duly carried.

Prof. J. C. Booth receiving the greatest number of votes, was duly elected President.

For Vice Presidents.—The following gentlemen received the greatest number of votes:

1. James H. Stebbins, Jr., } Three Local Vice-Presidents.
2. A. R. Leeds, }
3. C. F. Chandler, }
4. Arno Behr,
5. P. Schweitzer.
6. N. T. Lupton.

Mr. Casamajor then moved that the resolution preventing members who have read papers before the Society, from publishing them through any other source except the Journal of the American Chemical Society, for the space of thirty (30) days, be rescinded. This being seconded, was duly carried.

The following gentlemen were then elected to fill the remaining offices of the Society:

Corresponding Secretary—P. Casamajor.
Recording Secretary—Thomas S. Gladding.
Treasurer—T. O'C. Sloane.
Librarian—G. A. Prochazka.
Curators—William Rupp.
Committee on Papers and Publications—E. Waller, C. A. Doremus, L. H. Friedburg.

Committee on Nominations.

Board of Directors.—It was then moved and seconded that Drs Alsberg and Geyer, be elected Directors, in lieu of C. F. Chandler and James H. Stebbins, Jr.

James H. Stebbins, Jr., Geo. A. Prochazka,
A. R. Leeds, E. Waller,
C. F. Chandler, M. Alsberg,
P. Casamajor, H. Morton,
T. S. Gladding, Wm. E. Geyer,
T. O'C. Sloane, Wm. M. Habirshaw,
H. Endemann.

Mr. Casamajor then moved that this meeting be declared adjourned till the usual night for converzatione. This being seconded was duly carried. After which the meeting adjourned.

James H. Stebbins, Jr.,
Recording Secretary.

At the adjourned meeting, held Dec. 15th, 1882, no quorum being present, official business could not be transacted. A paper on the manufacture of tartaric acid was read by Dr. L. H. Friedburg. Mr. Percy Newman was nominated by E. Waller, Jas. H. Stebbins, Jr., and A. H. Elliott.

ON THE MANUFACTURE OF TARTARIC ACID.

By L. H. Friedburg, Ph. D.

In this country tartaric acid is hardly manufactured for its own sake but its preparation is unavoidably attached to the manufacture of cream of tartar. Here the starting points for tartaric acid are sablons, waste liquids and residues of different kinds, which render an analytical control troublesome, so that partly because of this, partly because of great dilution, the raw material is treated more or less empirically, after the known and often described methods * † with chalk and chloride of calcium or gypsum.

Abroad, the manufacture of tartaric acid is not everywhere a mere appendix to cream of tartar manufacturing, but forms an independent branch of manufacture. In such cases the raw material consists either of argols or of dry sablons or of lees. A careful analytical test has to be made before treating either of these mother substances, and the manufacture has to be carried on with the greatest care in order to avoid loss.

Until very recently these tartaric acid factories worked generally after the old plan as indicated above, viz : treatment with chalk and chloride of calcium or gypsum.

But it has to be recorded, and I will briefly do so in the following pages that a very neat and new, patented process, which according to my own experience is commendable, is now also in use.

This process is based on the decomposition of the mother substances, as named above, by slaked lime instead of chalk.

This preparation has been hidden in the European patents ‡ § under the heading, "Methods of obtaining the potassium in the form of hydrate, while making tartaric acid out of argols."

This heading is practically speaking untrue, because, as we shall see later on, the potassium is not finally gained as hydrate, though this is in the course of treatment formed and then transformed into sulphate or chloride.

The chemical process, which takes place in decomposing the bitartrate of potash in any mother substance into tartrate of lime,

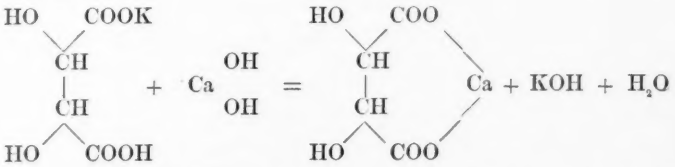
*Bericht ueber die Entwickelung der chem. Industrie; A. W. Hofmann. Vol. II, page 418, etc.

†Journal of the Society of Arts ; Robert Warington, Vol. xxiv, No. 1217, page 366, etc.

‡Die chemische Industrie ; Dr. Emil Jacobsen ; 1879, pages 86 and 238.

§Berichte d. deutschen chemischen Gesellschaft ; 1879, page 1366.

by means of slaked lime, is very simply conveyed through the following equation :



The practical difficulty which stood for so long a time in the way of realizing this decomposition for manufacturing purposes, was the difficulty of making the products of decomposition easily filterable. This the patentees have really overcome, and the method of working is smooth and goes like clock work. Slaked lime, freed from coarse pieces is taken in necessary quantities and a milk, not too thin prepared therefrom. This is heated to boiling, and argols, etc., in necessary quantity, are very gradually and in a state of finest powder, introduced into the boiling mass. The charging finished, boiling has to continue for two hours, the condensing steam being enough to keep the mixture in a concentrated form. Hydrate of potash and neutral tartrate of lime are formed in this way. The nitrogenous organic impurities of the raw materials are by the combined action on them, of lime and hydrate of potash, decomposed so as to form ammonia gas which is volatilized with the steam.

Boiling done, which takes place in an iron tank, the mixture is diluted by enough cold water and then the potash is neutralized by either muriatic or sulphuric acid. The process is finished with the help of litmus paper. Here the ammoniacal exhalations are to be considered, so as not to disturb the reaction.

The decomposition as described above takes place under constant stirring by means of an iron stirrer run by machinery.

After the formation of either chloride or sulphate of potash, the mass is still more diluted with cold water and stands best over night, stirring going on continuously, filtration then taking place the next morning. Here filter presses are used to great advantage. It is advisable not to use too high pressure, so as to get a soft cake, which can more easily be washed out, in order to get rid of the mineral potash salts. These latter are either boiled down, as long as the strength of the solution makes it pay, or they might* be treated with chloride of lime [bleaching powder] and thus trans-

*In case chloride of potassium was formed.

formed into chlorate of potash, which I advise manufacturers to try.

The cakes of brown tartrate of lime, which begin to exhale putrid odors by standing too long in a warm place, (in summer time six hours standing often will show this result) have speedily to be decomposed by sulphuric acid. This decomposition takes place in a wooden, lead lined tank, with heavy wooden stirrer moved by steam. The decomposition takes place in the cold and its completion is determined easily by methyl violet test paper.* No analysis has to be made here, if good paper is at hand, which allows one to guide the reaction so as to get the necessary or the excess of sulphuric acid wished for.

The brown solution of tartaric acid is filtered through filter-presses into wooden receivers.

It is not advisable to evaporate this acid down to the point of crystallization, because it contains impurities enough to spoil the mother liquors at a too early stage. If the course of manufacturing demands a readier transformation of raw material into money, this crude acid solution might be concentrated in the leaden pans to the right concentration for crystallizing or for precipitation by the stirring process, which we shall deal with on another page.

It is preferable to reprecipitate this acid as tartrate of lime, finishing the reaction with chalk and using litmus test paper.

The tartrate of lime thus obtained is filtered on a vacuum filter or by centrifugal power. Of course washing takes place, though slightly. This tartrate of lime is crystalline, light greenish-yellow, keeps perfectly well for any length of time required without decomposing.

It is decomposed in an apparatus similar to the one used for decomposition of the first brown tartrate of lime, by sulphuric acid, in the cold and the reaction finished with the aid of methyl violet test paper. The filtration of the very white gypsum thus obtained cannot be done through filter presses but has to take place on a vacuum filter, very thorough washing being required.

The tartaric acid solution thus obtained ought to stand between 12° and 14° Bé. It is ejected into the lead pans, evaporated at 80° C to the necessary density, by which dissolved gypsum is precipitated, run into the crystallizing boxes and let stand for crystallization. The crop of brown crystals is redissolved to a liquid of the

*Journal of the American Chemical Society; T. O'Connor Sloane, Vol. IV, Nos. 1-4, page 31, etc.

density 25° Bé and treated with bone black, which has been purified by muriatic acid, (so as not to leave a trace of phosphates,) at a medium temperature and under stirring.

The discolored liquid is run through a filter-press and thence into special lead pans. It is evaporated down to about 39° to 40° Be and run into lead boxes for crystallization.

The crystallization being a comparatively slow process, this liquid may be run into a proper tank with stirrer, stirred for several hours, thus yielding a crop of small crystals right away.

Either crystals are washed and dried in centrifugals, by using steam for washing.

The liquid running off from the first crystallization yields after evaporation another crop of white crystals. Then it becomes a brown mother liquor.

The mother liquors of the brown crystals can, under careful attention, be carried along through the sixth or seventh crystallization. Then the predomination of sulphuric acid and impurities does not allow further crystallization.

The mother liquors at that stage are diluted to a proper density, the greater part of the sulphuric acid removed by addition of slacked lime milk and the filtered liquid has then principally to be freed from iron salts and from phosphate of alumina.

The iron is easily expelled by taking care to keep it in the form corresponding to the protoxide, the presence of the phosphate of alumina makes it necessary to treat the liquids boiling with milk of lime, thus precipitating phosphate of alumina and forming an acid tartrate of lime, which is soluble. It has to be filtered hot and is decomposed by an addition of sulphuric acid, thus yielding very pure solutions of tartaric acid.

If a transformation of the acid thus gained, into bitartrate of potash should be wished for, which hardly would be prudent, the simplest way of arriving at this end would be the following:

The solution is divided into two equal parts, one-half *saturated* by caustic or carbonate of potash, so as to form a neutral tartrate of potash and then the other half added for the precipitation of the bitartrate.

New York, 15th December, 1882.

ON THE ACTION OF PHTHALIC ANHYDRIDE UPON GALLIC ACID.

By George A. Prochazka.

Mr. Stebbins* appears to have entirely misunderstood the gist of my remarks at the November meeting.

More than a year ago I experimented upon the action of phthalic anhydride upon gallic acid, with results similar to those of Mr. Stebbins. My object was to find a more economical method for the production on a large scale of gallein (and coerulëin) than given in the books. The substitution of gallic acid in place of pyrogallol readily suggested itself. Subsequently I found in a snyopsis by Ch. Lauth of the report on dye-stuffs, of the jury of the Paris International Exhibition of 1878, (Monit. Teint. 1878) that gallein was prepared by heating together phthalic anhydride and pyrogallic or *gallic* acid. My own results had been anticipated. My own results were never published. In making my remarks at the November meeting it was not my object to substitute a doubtful claim of originality,' substantiated by experiments hid under the bushel, in place of Mr. Stebbins,' but to call attention to the fact that he had been anticipated as early at least as 1878 by those European manufacturers, who had utilized the reaction in question. Mr. Stebbins does not appear to have been aware of this fact, which through a small notice had become known only to the initiated few. The very careful and detailed experiments (much more exhaustive than my own) of Mr. Stebbins, merit the widest attention, and the public owes to him a debt of gratitude for their publication.

*Journal Am. Chem. Soc. IV., 248.

INDEX OF AUTHORS.

A

Aillaud, M., Water from Panama, 242
Andre, G., Magnesium oxychlorides, 230
Andre, G., Oxychloride of zinc, 239
Armstrong, H. E., laws of substitution etc., 206
Atkinson, R., & H. Yoshida. Menthol etc., 144

B

Baeyer, A. Combination in the indigo group, 175
——Syntheses by phenylacetylene, 176, 206
Barr, K. L., Precipitation of alums by sodic carbonate, 258
Bartoli, B., Synthesis of organic compounds, 239
Baubigny, H., Action of sulphides on nickel salts, 239, 240
Bechamp, A., H_2O_2 on the coloring matter of blood, etc., 240
Becker, P., Monophenyl-boro-chloride etc., 206
Behr, A., Crystallized anhydrous grape sugar, 11
Benthsen, A., Normal dithiurethanes, 268
Bert, P., Action of hydrogen peroxide, etc., 239
Berthelot, M., Absorption of gases by platinum, 239
——Decomposition of mercury salts, 233, 234
——Decompositions of silver salts etc., 235
——Double salts by fusion, 239
——Double salts of mercury, 208
——Electrolysis of H_2O_2, 240
——Double salts of Mercury, 231
——Sulphuretted ureas from carbon oxysulphide, 236
—— Union of free hydrogen with ethylen, 235
Bevan, E. J., & C. F. Cross, Apparatus for determining melting points, 169
—— Bast fibres, 169
Birubaum, K., Iodine on silver salts of aromatic series, 264
Bloxam, W. P., Crystallization from solutions of salts etc., 261
Bœhringer, C., Cinchotin, etc., 267
Boisbaudran, L de., Crystallized gallium oxy-chloride, 233
——Separation of gallium, 237, 238, 239, 240, 242, 273
Bollert, A., Anthramin, 207
Bourgoin, E., Potassium cyanide on the trichloracetate, 231
Bourgougnon, A., Analyses of calculi, etc., 215
Brauner, B., Chemistry of cerium metals, 176
——Chemistry of rare earth metals, 169
——On Didymium, 240
Brenneman, A. A., Review of industrial chemistry, 72
Bruehl, J. W., Physical constants, etc., 264
Brunner, H., Preparation of coloring matter, etc., 205

C

Cailletet, L., Liquified ethylene for low temperatures, 238
Cahours, A., Formation of fatty acids etc., 233
Calm, A., Amidated naphthalines from naphthols, 390
Carnelly, T., Action of heat on mercuric chloride, 225
Casamajor, P., Note on asbestos filters, 248
——Volumetric estimation of copper and lead, 35
Cazeneuve, C., Dichloride of camphor, 233
Chancel, G., Nitrogenized acids from acetones, 208
Chapotreant, P., On the gastric juice, 242
Chapuis, G., Liquefaction of ozone, 238
——Pernitric acid, etc., 237
Chastaing, M., Morphine, 76
——Pilocarpin, 78

——Action of acids on pilocarpin, 236
Chautard, P., Oxidation of pyrogallol, etc., 237
Claisin, L., Action of haloid acids on HCy, 221
Claus, A., Bases from addition of chinolin aldehydes, etc., 265
—— Dichlornaphthalin etc., 229
——PCl_5 on Benzoyldiphenylamine, 264
——PCl_5 on Naphthol sulphonic acid, 228
——Synthesis of Homologues of anilin, 228
Clermont, P de., Oxidation of Pyrogallol, etc., 237
C eve, T., Preliminary note on didymium, 240
Cleves, P. T., Remarks on didymium, 240
Colson, A., On Silicium, 240
——New compounds of C and Si, 237
——Action of CS_2 on Si, 240
Combes, M., On the compound NH_3, 240
Conrad, M., Chloromalonic acid, etc., 270
Cowper, R., Analysis of oxidized iron from condenser, 219
——Solubility of glass, etc., 219
Crafts, J. M. & C. Friedel, Decompositions produced by aluminium chloride, 170
Cross, C. F. & E. J. Bevan, Bast Fibres, 169
————Apparatus for determining melting points, 169
Cross, C. F. & A. Higgin, Reaction of chromic anhydride with sulphuric acid, 170

D

Damm. G., Resorcin dyes, 268
Darton, N. H., Estimation of Tannic acid in tanning materials, 4
——Comparative value of methods for determining tannic acid, 49, 62
——Convenient form of weighing flask, 6
David, J., Determination of glycerin, 239
Debray, H., Explosible alloys of zinc, etc., 240
——Some reactions of $Hg Cl_2$, 238
Dehue, M., Dichlornaphthalin, etc., 229
Demarcay, E., Formation of fatty acids, etc., 233
——Vaporizing metals in vacuo, 242
Deml. W., Bodies combustible with difficulty, 269
Deville, St. C., Explosible alloys of zinc, etc., 240
Dietzell, B. E, Evolution of free nitrogen in fermentation, 267
Ditte, A., Acid solutions on iron protoxide, 234
——Action of alkaline solutions on Sn O, 235
——Decomposition of lead salts, 237
——Potassa on lead oxide, 237
Doebner, O., Combinations of benzotri chloride, 207
Dogiel, J., Effects of arsenic, 268
Donald, G. & E. J. Mills, Action of oxides on salts, 142
Doremus, C. A., Composition of elephants' milk, 157
Duclaux, E., Gastric digestion, 234
Dumreicher, O. V., Action of aluminium chloride on monobrombenzol, 174
Duvillier, E, Combinations in creatin groups, 273

E

Elliott, A. H., Nitro-saccharose, 147
———— " (a correction), 186
Elliott, A. H. & F. Sands, Notes on bone oil, 153
Erlenmeyer, E., Halogenized organic acids, etc., 175
Etard, M., Isomery of cuprous sulphites, 241

F

Fenton, H. J. H., Transformation of urea, 221
Filhol, E, Neutral arsenates, 272
Fincke, H., Action of amines on chinones, 226
Fischer, E., On caffein, 174
——Transformation of xanthin, etc., 264
Fischer, O., Nicotinic acid from pyridin, 176
Flawitzky, F., Rotation in the plane of polarization, etc., 173
——Molecular power of refraction of terpenes, 173
Flight, W., Composition of alloys, 171
——Action of Sodium hydrate, etc., on feldspars, etc., 199
Flosray, M., Double salts by fusion, 239
Foerster, K., Jorissen's reaction for fusel oil, 207

Foerster, K., Furfurol in fermented liquids, 229
Foerster, P., Identity of coloring matter with quercitrin, etc., 206
Forcraud M. De., Hydrate of hydrogen sulphide, 242
Forst, C., Cinchotin, etc., 267
Friedburg, L. H., Bisulphide of carbon, 252
—— Manufacture of Tartaric acid, 292
Friedel, C. & J. M. Crafts, Decompositions produced by aluminium chloride, 170
Friedlander, P., Carbostyril, 230
Friese, G., Normal dithiurethanes, 268

G

Gantter, Fr., Bromo substitution products of sebacylic acid, 204
Gautier, A., Insoluble modification of pepsin, 237
Gerber, M., Rosanilin, 237
Girare, A., Det. of astringent matters in wine, 242
Gladding, T. S., Determination of reverted phosphates, 113
—— Estimation of phosphoric acid, as magnesium pyrophosphate, 135
Gladding & Stillwell, Acetate of lime, 94
Gladstone, J. H. and A. Tribe, aluminium alcohols, 142
Gleechmann, L., Dimethyl-phenyl-phosphine, reaction of, 206
Goldschmidt, H., Phosphorus pentasulphide, 227
Goldschmidt, H. & V. Meyer, Density of gases, 176
Gorgen, A., Basic salts of manganese, 242
Griess, P., Cyanogen on picrammic acid, 264
Grimann, E., Bromine on quinolein and pyridin, 242
Guinochet, Aconitates, 231
Guthzeit, M., Chloromalonic acid, etc., 270

H

Haas, B., Determination of sulphurous acid in wine, 205
Haller, A., Camphol-urethane, 235
Haller, M. A., Carbonic ether of borneol, 76
—— Essential oil of savory, 77
Hallock, A. P., Direct estimation of H_2S in gas, 177
Hartley, W. N., Chemistry of cerium compounds, 203
Hartley, W. N., Photographs of spectra, etc., 169
—— Spectra of carbon compounds, 144
—— Rabdophane, a new mineral, 203
Hautefeuille, P., Liquefaction of ozone, 238
—— Pernitric acid, etc., 237
Heckel, E., Kola nut, etc., 234
Hell, C., Bromosubstitution products of sebacylic acid, 204
—— Combination of C with S and Br., 226
Henninger, A., Glycol in wine, 242
Heumann, K., Chlorosulphurous acid with metalloids, etc., 261
Higgin, A., Dibenzoylaniline, etc., 171
Higgin, A. & C. F. Cross, Reaction of chromic anhydride with sulphuric acid, 170
Hjelt, E., Bromine on allylmalonic acid, 270
—— Caprolactone, dilactones, lactones, 270
—— Oxypropylmalonic acid, 270
—— Water on isobromcapronic acid, 270
Hodgkin J. & D. Howard, Alkaloid from cinchona, 168
Howard, D. & J. Hodgkin, Alkaloid from cinchona, 168
Hummel, J. J., New compounds of Brazilein and Haematein, 260

J

Jacobsen, O., Bromchloral, chlorobromal, etc., 269
Jacquelin, M., Pure carbon for electric light, 235
Japp, F. A., Action of aldehydes on phenanthra quinone, etc., 198
—— Constitution of amarine and lophine, 225
Japp, F. R., Action of acetone on phenanthra-quinone, 221
—— Determining constitution of quinones, 199
Jean, F., Clarification of champagne wines, 234
—— Det. of tannic and œnogallic acid in wine, 233
Joannis, M., Heat from ferricyanhydric acid, 231
—— Heat of formation of HCy S, etc., 234
Joly, A., Saturation of phosphoric acid, 232
Jorissen, W. A., Furfurol mented liquids, 268

K

Kelbe, W., Capronic acid in resin oil, 228
—— Expulsion of the sulpho group, 175
Kiliani, H., On gum arabic, 174
Knecht, E., New isomer of orcin, 227
Kraut, K., On tropin, 264
Koechlin, P., Chlorosulphurous acid with metalloids, etc., 261
Konig, F., Succinic acid by fermentation, 205

L

La Coste, W., Halogen derivatives of chinolins, 268
——Reactions of addition products of chinolin, 206
Ladenburg, A., History of atropin, 176
Ladureau, A., Phosphoric acid in soils of France, 77
Landsberg, L., Syntheses by phenyl-acetyene, 176, 206
Lapraik, W., Spectroscopic study of chlorophyll, 257
Lau, M., Effects of compression on the hardness of steel, 235
Laubenheimer, A., Orthodinitro compounds, 269
Laur, P., Reduction of silver ores, 241
Lebon, G., Two new antiseptics, 242
Leeds, A. R., Acrolein urea, 59
——Contamination of the New York water supply, 127
——Diphenylamine-acrolein, 32
——Reduction of metallic oxides in sunlight, 3
Leuckart, R., action of mono-brom-cinnamic acids with conc. sulphuric acid, 173
Lewes, V., Experiments on potassium tetra and pentathionate, 224
Liebermann, C., Anthramin, 207
——Azoanthrol dyes, 266
Liebermann, L., Determining melting points of metals, etc., 263
——Sulphurous acid in wine, 263
——Distillation of tartaric acid, 263
Liebmann, A., Synthesis of homologous phenols, 205
Link, G., Analyses of nephrites from lake dwellings, 206
Lloyd, F. J., Estimation of retrograde phosphates, 224
Losanitsch, S. M., CS_2 on P-nitranilin, 264
——Nitric acid on tribromanilin, 264
Lunge, G., Nitrogen tetroxide with sulphuric acid, etc., 265
——Nitrous anhydride as vapor, 266

M

Mailfert, M., Researches on ozone, 235, 237
Manumene, E. G., Synthesis of quinine, 236
Mallet, J. W., Fractional dehydration of ammonium alum and the atomicity of aluminium, 180
——Properties of pure metallic aluminium, 145
Maquenne, M., Action of ozone on manganese salts, 234
Meldola, R., Aromatic derivatives of methane, 202
Mentschukin, N., Etherification of the oxacids, 205
Merling, G., Tropin, 226
Meyer, R., Constitution of cumic acid, 266
——, Tetrabrombenzol, 175
Meyer, P. T., Mustard oil glycolide, 267
Meyer, V., Lecture experiments, 227
Meyer, V. & H. Goldschmidt, Density of gases, 176
Michaelis, A., Mono-phenyl-boro-chloride, etc., 206
——Tolyl-methyl-keton, 206
Mills, E. J. & G. Donald, Action of oxides on salts, 142
Mills, E. J., & J. Pettigrew, Steeping of barley, 143
Morin, H., Essential oil of Licari Kanali, 232
Morley, H. F., Oxypropyltoluidine, 206
Morris, G. H., Some constituents of resin spirit, 200
Mueller, E., Constitution of cumic acid, 266
Muir, M. M. P., Action of water on BiI_3, 142
——Volumetric estimation of bismuth, 141
Mulder, E., Normal cyanic acid, etc., 176
——Thermometric knowledge of ozone, 266
Muller, C. L., Halogenized organic acids, etc., 175
Muntz, A., Galactin, 231

N

Neumeister, R., Bromchloral, chlorobromal, etc,, 269
——Bromdichloracetic acid, etc., 269
Nichol, W. W. I., Action of heat on thioformanilide, 206

O

Oehler, H., PCl_5 on naphtholsulphonic acid, 228
Ogier, J., New oxychloride of sulphur, 231
——Sp. gr. of pyrosulfuryl chloride vapor, 78
Osmond, F., Vanadium from basic slags, 241
Ostermaier, H., Carbostyril, 230
O'Sullivan, C., Gamma and beta-amylan, 143
Otto, R., Synthesis of alkyl-disulph oxides, 176

P

Palewski, B., Critical temperatures, 264
Papasogli, G., Synthesis of organic compounds, 239
Parmentier, F, Silico-molybdic acid 77
Pellet, H., Antiseptic properties of salicylic acid, 238
Perkin, A. S., New compounds of Hæmatein and Brazilein, 260
Perkin, W. H. Action of acetyl chloride on fumaric acid 221
——Luminous incomplete combustion of ether, etc., 259
——Rotary polarization of chemical subst. 257
Pettigrew, J. & E. J. Mills., Steeping of barley, 143
Phillip, J., On tungsten bronzes, 266
Pictet, A., Quinolein and lutidin, 272
Pinner, A., Condensation of acetone, 268
Plagemann, A., Amines on dichlornaphthachinone, 265
Ponomareff, J., Cyanic and cyanuric ethers, 267
Prochazka, G. A., Action of phthalic anhydride on gallic acid, 296

R

Radcliffe, T., Rock salt from Saltville, Va., 255
— Deposit of zinc oxide in a blast furnace, 256
Regnard, P., Action of hydrogen peroxide, etc., 239
Reinherz, H., Iodine on silver salts of aromatic series, 264
Reinke, J., Reducing properties of living cells, 176
Renard, A,, Products of distillation of colophony, 233
Rennie, E. H., Action of ethyl chlorocarbonate on benzene, 143
——Benzyl-phenol, etc., 143, 204
Reynolds, J. E., Apparatus for liquefying ammonia, 220.
Ricciardi, L., Composition of ashes from Vesuvius, 237
——Composition of the banana, 272
Robinet, E., Antiseptic properties of salicylic acid, 238
Robinson, H. H., Constitution of amarine and lophine, 225
Roemer, H., Anthra cylamin, 230
Roscoe, H., Atomic weight of carbon, 237
——Earth-metals in Samarskite, 222
——Spectrum of terbuim, 223
Rosenfeld, M., Lecture experiments, 205
Rosenstiehl, A, Rosanilin, 237
Roser, W., Terebenthenic acid, 227
Rousseau, G., Chloroform on beta-naphthol, 241
——Diatomic alcohol from beta-naphthol, 77
Russell, W. J., Spectroscopic study of chlorophyll, 257

S

Sacc, F., Cucurbitaceæ of Uruguay, 236
——F., Products from Uruguay, 237
Sakurai, J., Metallic compounds containing bivalent hydro carbon radicals, 258
Sands, F. and A. H. Elliot, Notes on bone oil, 153
Sarauw, A., Reaction of phosgen on diaze, etc., 175
Seubert, K., Analyses of Nephrites from lake dwellings, 206
Schuster, A., Spectrum of terbium, 223
Schulze, K. E., Phoron from glycerin, 176
Schutzenberger, P., On silicium, 240
Schreiner, L., Resorcin dyes, 258
Schotten, C., Piperidin, 262
Schwanert, H., Large crystals in urine, 174
Sloane, T. O'C., Fat extracting apparatus, 250
——Methyl-violet test paper, 31

Spring, W., Chlorine on sulphonic compounds, etc., 263
——Formation of alloys by pressure, 269
Smith, W., and T. Takamatke, Pentathionic acid, 199
——B. E., Preparation of diethylnaphthylamine, 200
——Sulphuric acid on diethyl naphthylamine, 201
——Phosgen gas on diethyl naphthylamine, 201
Stebbins, Jr., J. H , Action of phthalic anhydride on gallic acid, 244
——Artificial indigo, 81
——Laboratory notes, 214
Stillwell & Gladding, Acetate of lime, 94
Stone, G.C., Determination of Zinc as pyrophosphate, 26
Streatfield, F. W., Action of acetone on phenanthraquinone, 221
——Action on phenanthraquinone, 198
——Determining constitution of quinones, 199

T

Takamatke, T. and W. Smith, Pentathionic acid, 199
Thenard, P., Black phosphorus, 273
Thomsen, J., Constitution of Benzol, etc., 229
——Optical rotation of malic acid, etc., 263
——Refraction and heat of combustion, 176
Thomson, J. M., Crystallization from solutions of salts, etc., 261
Thorpe, T. E., Action of oxychlorides of sulphur on silver nitrate, 223
——Action of thiophosphoryl chloride on silver nitrate, 224
——Reducing agents with ferric salts, 223
Thresh, J. C., Examination of Buxton thermal water, 170
Tribe, A., and J. H, Gladstone, Aluminium alcohols, 142
Traube, M., Rendering oxygen active, 207
Troost, L., Iodides of phosphorus, 272
——New ammonium nitrates and acetates, 234

U

Urech, F., Combination of C. with S. and Br. 226

V

Van der Mulen, H. G. L., Thermometric knowledge of ozone, 266
Venable, F. P., Alcoholic potash on heptylene bromide, 254
——Heptylene from heptane of P. Sabiniana, 22
Veley, V. H., Higher oxides of manganese, etc., 168

W

Wait, C. E., An earthy ferric sulphate, 61
Wallach, O., Azo dyestuffs, 173
Wallach, O., Basic compounds from acido-amides, 206
Waller, E., Determination of phosphorus in iron ores, 88
——Laboratory notes, 212
——Review of analytical chemistry (proximate), 189
——Review of analytical chem. (mineral), 160
——Water supply of the city of New York, 15
Waltz, G., Propyl and isopropyl-succinic acid, 270
Warrington, R., Determination of nitric acid, 258
Warth, C., Capronic acid in resin oil, 228
Wethered, E., Composition of Pennant grits etc., 144
Widmann, O., Synthesis of thymol, 205
Wietzky, R., Naphthyl-sulphuric acid 227
Will, W., Bodies produced from sulpho-carbanilid, 230
Wills, E. J., Precipitation of alums by sodic carbonate, 258
Winssinger, C., Chlorine on sulphonic compounds etc., 263
Witz, G., Vanadium from basic slags, 261
Wroblenowski, M., Composition of hydrated carbonic acid, 236
Wurtz, A., Ethylenic chlorhydrin on quinolein etc., 272

Y

Yoshida, H. & R. Atkinson, Menthol etc., 166

Z

Zenger, V., Physical constants, etc., 264
Zimmermann, J., Chloracetic acid ether on phenylendiamin, 267
Zincke, T., Amines on chinones, 265

INDEX OF SUBJECTS.

A

Acetate of lime, its manufacture and analysis, Stillwell & Gladding, 94
Acetic acid, preparation of, (pat)., 41
Acetone, condensation of, 268
Acetone on phenanthraquinone, 221
Acetones, nitrogenized acids from, 208
Acetyl chloride, action on fumaric acid, 221
Aconitates, 231
Acrolein-urea, A. R. Leeds, 59
Alcohols aluminium, 142
Alcohol diatomic, from beta-naphthol, 77
Alcohol from potatoes, (pat), 41
Alcohol, manufacture of, (pat), 40
Alcohol rectifying, (pat), 47
Alcohol removing bad smell and taste from, (pat), 43
Aldehydes, action on phenanthra-quinone, 198
Aldehydes, bases from addition of, etc., 265
Aldehyde and ammonia reaction for determining the constitution of quinones, 199
Alizarine, preparation of artificial, (pat), 40
Alkalies, pure caustic by electrolysis, (pat), 110
Alkylbromides, in synthesis of anilin homologues, 228
Alkyldisulphoxides, synthesis of, 176
Alloys ancient, composition of, 171
Alloys and metals, determining melting points of, 263
Alloys formed by pressure, 269
Allylmalonic acid, bromine on, 271
Alum, ammonium, fractional dehydration of, etc., J. W. Mallet, 180
Alums, precipitation by sodic carbonate, 258
Aluminium alcohols, 142
Aluminium, atomicity of, etc., J. W. Mallet, 180
Aluminium chloride action of, 174
Aluminium chloride, decomposition by, 170
Aluminium pure metallic, J. W. Mallet, 145
Aluminium sulphate, (pat), 46, 111
Aluminium sulphate, separation of iron from, (pat), 40
Amarine and lophine, constitution of 225
Amides acido, Basic compounds from 206
Amines on chinones, 226, 265
Ammonia, apparatus for liquefying, 220
Ammonia from elution lyes, (pat), 46
Ammonia from molasses swill (pat), 112
Ammonia from purifiers of gas-works, (pat), 47
Ammonia, new combinations with nitric and acetic acids, 234
Ammoniacal waste waters purification, (pat), 42
Ammonium alum. Fractional dehydration, etc. J. W. Mallet, 180
Ammonium sulphate from peat, (pat), 41
Amylan, Gamma & Beta, 143
Analytical chemistry, review of, 160
Analytical chemistry, (proximate) review, 189
Anilin, synthesis of homologues of etc., 228
Anthracylamin, 230
Anthramin, 207
Antiseptics, two new, 242
Aromatic series, iodine on silver salts 264
Arsenates, neutral, 272
Arsenic, effects of, 268
Asbestos filters, by P. Casamajor, 248
Ashes from Vesuvius, composition, 237
Astringent matters in wine det. of 242
Atropin, history of, 176
Azo anthrol dyes, 266
Azo dyestuffs, 173

B.

Banana, ripe and unripe, composition of, 272
Barium sulphate. Laboratory note, E. Waller, 212

Barley, steeping of, 143
Battery Galvanic, (pat), 41
Benzene, action of ethyl chlorocarbonate on, 143
Benzoic acid, etc., preparation of, (pat), 40
Benzol, constitution of, 229
Benzol tetra-brom, 175
Benzo trichloride combined with aromatic bases, 207
Benzoyl diphenylamine, etc., PCl_5 on 264
Benzyl-phenol and its derivatives, 143, 204
Bismuth iodide, action of water on, 142
Bismuth volumetric estimation of, 141
Bisulphide of carbon, L. H. Friedburg, 252
Blue coloring matters, preparation of, (pat), 40, 42
Bodies combustible with difficulty, 269
Boiler incrustation preventing, (pat), 39
Bone oil, notes on, A. H. Elliott and F. Sands, 153
Borneol carbonic ether of, 76
Brazilein and Hæmatein new compounds of, 260
Bromchloral, bromochloroform, etc., 269
Bromdichloracetic acid, etc., 269
Bromide, heptylene, alcoholic potash on, F. P. Venable, 254
Bromine, expulsion of sulpho group by, 175
Bromine on quinolein and pyridin, 242
Bromine and S. combination with C., 226
Bronze phosphor-lead, (pat), 44

C

Caffein, 174
Calculi, analyses of, A. Bourgougnon, 215
Camphol urethane, 235
Camphor Dichloride, 233
Camphor peppermint, and its derivative, 144
Catprolactone, 270
Capronic acid in resin oil, 228
Carbanilid Sulpho derivatives, 230
Carbon atomic weight of. 237
Carbon compounds, spectra of, 144
Carbon, combination with S. and Br., 226
Carbon and Silicium, new compounds of, 237
Carbon Bisulphide, L. H. Friedburg 252
Carbon Disulphide on Silicium, 240
Carbon Disulphide on P-nitranilin, 264
Carbon oxydichloride, action on diethylnaphthylamine, 201
Carbon oxysulphide, transformation to sulphuretted ureas, 236
Carbon, pure for electric light, 235
Carbonic acid hydrated, composition of, 236
Carbostyril, 230
Cements, etc., method of testing, (Pat) 42
Cerium compounds, 203
Cerium metals, chemistry of. 176
Chemistry, Industrial, review of, A. A. Breneman, 72
Chinolin, reactions of addition products, etc., 206
Chinolin, Halogen derivatives of. 268
Chinones, Amines on, 226, 265
Chloracetic acid ether on phenylendiamin, 267
Chlorates, (Pat.), 79
Chlordibrom acetic acid, etc., 269
Chlorine, action on sulphonic compounds, etc., 262
Chlor-naphthol, etc., 229
Chloro bromal, chloro bromoform, etc., 269
Chloroform on beta naphtol, 241
Chloromalonic acid and derivatives, 270
Chlorophyll, spectroscopic study of, 257
Chlorosulphurous acid with metalloids, etc., 261
Chromic anhydride reaction with sulphuric acid, 170
Chromium, compounds for tanning, (Pat.) 48
Cinnamic acids, mono-brom, action, 173
Cinchona Bark, new alkaloid from 168
Chichotin, etc., 267
Coal, fractional distillation of, (Pat.), 111
Coffein, etc., from xanthin, 264
Cold, generating, (Pat.), 109
Colophony, products of distillation of, 233
Coloring matters, (Pat.), 40, 42, 44, 46, 48, 80, 108, 110
Coloring matters of the Rosaniline group, (Pat.) 46
Coloring matter, preparation by nitro substances, etc., 205

Combustion, Refraction and heat of, 176
Constants, Physical, etc., 264
Cooling apparatus, (Pat.), 39
Copper and Lead, volumetric estimation of, P. Casamajor, 35
Copper refined from mattes (Pat.), 79
Correction Nitro saccharose, A. H. Elliott, 186
Creatin Groups, combinations in, 273
Crystallization from solutions of compound salts, 261
Cucurbitaceae of Uruguay, 236
Cumic acid, constitution of, 266
Cuminol, Synthesis of Thymol from, 205
Cuprous Sulphites, Isomery of, 241
Cyanamide from urea, 221
Cyanic acid and derivatives, 176
Cyanic and cyanuric ethers, 267
Cyanogen on picraminic acid, 264

D

Depilatory for hides, (Pat.), 109
Diallylacetic acid, HBr. and Br. on, 271
Diazotizing for formation of coloring matters, (Pat.), 46
Dibenzoylaniline and its isomerides, 171
Dichlor-naptha-chinone, amides on, 265
Dichlornapthalin, etc., 229
Didymium, 240
Diethylnapthylamine preparation, 200
Diethylnapthylamine action of sulphuric acid on, 201,
Dilactones, 271
Dimethyl-phenyl phosphine on ethylene bromide, 206
Diphenylamine-acrolein, A. R. Leeds, 32
Disinfecting, etc., by carbonized peat coal, (Pat.), 48
Dithiurethanes normal, 268
Dyes, Blue and Violet, (Pat.), 110

E

Earth metals in Samarskite, 222
Earth metals, rare, 169
Elephants' milk, composition of, C. A. Doremus, 157
Emery, wheels, (Pat), 41
Ether etc., incomplete combustion of, 259
Etherification of the oxacids, 205
Ethers, cyanic and cyanuric, 267
Ethylen, union of free hydrogen with, 235
Ethylen, liquefied for low temperatures, 238
Ethylene bromide, reaction on dimethyl phenyl phosphine, 206
Ethylenic chlorhydrin on pyridic bases etc., 272
Eupittonic acid, preparation from wood tar, (Pat), 40
Experiments, lecture, 205, 227
Explosive compounds, (Pat),41, 48, 80
Extraction, apparatus for analytical purposes, (Pat), 47

F

Fabrics, woven finish for, (Pat), 111
Fat extracting apparatus T. O'C. Sloane, 250
Feldspars, action of sodium hydrate etc. on, 199
Fermentation, evolution of free nitrogen in, 267
Ferric salts, behavior of reducing agents with, 223
Ferric sulphate, earthy, C. E. Wait, 61
Ferricyanhydric acid, heat from, 231, 233
Fertilizer, (Pat), 46
Fibres, Bast, chemistry of, 169
Filtering apparatus, (Pat), 47
Filters Asbestos, P. Casamajor, 248
Fumaric acid, acetyl chloride on, 221
Furfurol in fermented liquids, 229, 268
Furnace, blast, zinc oxide in, T. Radcliffe, 256
Fusel oil, Jorissen's reaction for, 207

G

Galactin, 231
Galena, analysis of, J. H. Stebbins, 214
Gallic acid, action of phthalic anhydride on, J. H. Stebbins, 244
Gallic Acid, action of phthalic anhydride on, G. A. Prochazka, 296
Gallium, oxychloride crystallized, 233
Gallium, separation of, 237, 238, 239, 242, 273
Gastric digestion, 234
Gastric juice, on the, 242
Gas, direct estimation of H_2S in, A. P. Hallock, 177
Gas, illuminating and heating, (Pat), 46
Gases, determining density of, 176
Gases, driving out of liquids etc., (Pat), 45

Gases from coke furnaces, utilizing, (Pat), 109
Glass, solubility in certain reagents, 219
Glucose, removing gypsum from, (Pat),108
Glycerin, determination of, 239
Glycerin, phoron from, 176
Glycol in wine, 242
Glycolide Mustard oil, 267
Goldmark, J., obituary, 7
Grits, Pennant, composition of, 144
Gum arabic, 174

H

Hæmatein and brazilein, new compounds of, 260
Hematosin etc , 240
Heptylene, from heptane of P. Sabaniana, F. P. Venable, 22
Heptylene bromide, alcoholic potash on. F. P. Venable, 254
Hides, preserving and waterproofing, (Pat), 45
Hydrocarbon radicals in metallic compounds, 258
Hydrocyanic acid, action of haloid acids on, 221
Hydrocinchonidin and hydrochinidin, etc., 267
Hydrogen, (Pat), 46
Hydrogen dioxide, electrolysis of, 240
Hydrogen peroxide, on coloring matter of the blood, 240
Hydrogen peroxide, action on organic matter, etc., 239
Hydrogen, sulphide, hydrate, 242
Hydrogen sulphide, on nickel chloride 240

I

Indigo artificial, J. H. Stebbins, 81
Indigo group, combination in the, 175
Iodine, on silver salts of aromatic series, 264
Iodo salicylic acids, etc.,
Iron, acid proof coating on, (Pat), 109
Iron protoxide, acid solutions on, 234
Iron, oxidized, from condenser, 219
Iso-brom-capronic acid, water on, 270

K

Kainit, (Pat), 43, 45
Kola nut, etc., 234

L

Laboratory notes, J. H. Stebbins, 214
Laboratory notes, E Waller, 212.
Lactones, etc., 270, 271
Lead acetate, laboratory note, E. Waller, 213
Lead and Copper, Volumetric estimation of, P. Casamajor, 35
Lead and Silver from Ores, (Pat). 43
Lead oxide Potassa on, 237
Lead salts, decomposition by alkalies, 237
Lead white, manufacture of (Pat). 39
Lime acetate, manufacture and analysis, Stillwell & Gladding, 94
Liquids, treating with gases, etc., (Pat). 47
Lophine and amarin, constitution of 225
Lubricants, (Pat)., 108
Lutidin and quinolein, 272

M

Magnesia, etc., from Stassfurt salts, (Pat) , 78
Magnesia, Preparation from dolomite, etc., (Pat)., 45
Magnesium oxychlorides, 230
Magnesium Pyrophosphate, estimation of phosphoric acid as, T. S. Gladding, 135
Malic acid, optical rotation of, 263
Manganese, higher oxides, etc., of, 168
Manganese, basic salts of, 242
Manganese salts, Ozone on, 234
Maltose, (Pat)., 109
Melting Points, Apparatus for determining, 169, 263
Menthol, and its derivative, 144
Methane, Aromatic derivatives of, 202
Metallic Compounds containing bivalent radicals, 258
Metalloids, Chlorosulphurous acid with, 261
Methylnaphthalene, dyes from, (Pat.) 111
Methyl Violet test paper, T. O' C. Sloane, 31
Metals, Vaporization in vacuo, 242
Mercuric chloride, Reactions, 238
Mercuric chloride, Action of heat on, 225
Mercury salts, decomposition by potassium salts, 233, 234
Mercury, double salts, 208, 231
Milk, Elephants, composition of, C. A. Doremus, 157
Milk, Tester, (Pat)., 108
Mono-phenyl-boro-chloride and derivatives, 206
Morphine, Functions, transformation and solubility, 76

Molasses swill, Distillation of, (Pat)., 112
Mustard oil, Glycolide, 207

N

Naphthalene, Coloring matters from nitro derivatives of, (Pat)., 46
Naphthalene series. Substitution in, 206
Naphtalines, Amidated from A and B naphthol, 270
Naphthol beta, Chloroform on, 241
Naphthol beta, Diatomic alcohol from 77
Naphthols, Conversion into monamines, (Pat)., 42
Naphthol sulphonic acid, PCl_5 on, 228
Naphthol sulphonic acid derivatives, 229
Naphthylsulphuric acid, 227
Nephrites, Analyses of, 206
NH_2, 240
Nickel chloride, Hydrogen sulphide on, 240
Nickel sulphate, Metallic sulphides on, 239
Nicotinic acid from pyridin, 176
Nitranilin P-, CS_2 on, 264
Nitric acid, Determined as nitric oxide, 258
Nitro benzaldehyde preparation (Pat). 109
Nitro benzylchloride for coloring matters, (Pat)., 48
Nitrogen free, evolved by fermentation, 267
Nitrogen from air, (Pat)., 44
Nitrogen tetroxide with sulphuric acid, etc., 265
Nitrogenized acids from acetones, 208
Nitro glycerine, Cotton and dextrine for, (Pat)., 80
Nitro-saccharose, A. H. Elliott, 147
Nitro-saccharose, A correction, A. H. Elliott, 186
Nitro substances, for preparing coloring matter, 205
Nitrous anhydride as vapor, 266

O

Oenogallic acid in wine determination, 233
Oil, Essential, of Licari Kanali, 233
Oil of savory, Essential, 77
Orcin, New isomer of, 227
Organic acids, Halogenized, etc., 175
Organic Compounds, synthesis by electrolysis, 239
Orthodinitro compounds, 269
Oxides, Action on salts, 142
Oxides, Metallic, reduction in sunlight, A. R. Leeds, 3
Oxygen from air, (Pat)., 47
Oxygen, Rendering active, 207
Oxypropylmalonic acid, etc., 270
Oxypropyltoluidin, 206
Oxysulphides, organic, etc., chlorine on, 26
Ozone Generator, (Pat)., 78
Ozone, Liquefaction of, 238
Ozone, On manganese salts, 234
Ozone, Researches on, 235, 237
Ozone, Thermometric knowledge of 266

P

Paraffine from crude paraffine without presses (Pat.), 43
Paranitro benzaldehyde (Pat.), 48
Patents, Foreign, 38, 108
Peat coal carbonized, for disinfecting, etc. (Pat.) 48
Pentathionic acid, 199
Peppermint camphor and its derivatives, 144
Pepsine, insoluble modification of, 237
Pernitric acid composition and atomic weight, 237
Phenanthraquinone, action of acetone on, 221
Phenanthraquinone, action of aldehydes on, 198
Phenol-acetylene, synthesis by, etc., 176
Phenol, benzyl and derivatives, 204
Phenols, homologous, synthesis of, 205
Phenyl-acetylene, syntheses by means of, 206
Phenylen diamin, chloracetic acid ether on, 267
Phoron from glycerin, 176
Phosgen, reaction on diazo amido compounds, 175
Phosgen gas, action on diethylnaphthylamine, 201
Phosphates, native basic in the soda manufacture (Pat.), 110
Phosphates, retrograde, estimation of, 224
Phosphates, reverted, determination of, T. S. Gladding, 113
Phosphoric acid, estimation as magnesic pyrophosphate, T. S. Gladding, 135
Phosphoric acid in soils of France, 77

Phosphoric acid, saturation by bases, 232
Phosphorus, black, 272
Phosphorus, determination in iron ores, E. Waller, 88
Phosphorus iodides, 272
Phosphorus pentachloride on naphthol sulphonic acid, 228
Phosphorus pentachloride on acetyl, etc., 264
Phosphorus pentasulphide, 227
Phthalic anhydride, action on gallic acid, J. H. Stebbins, 244
Phthalic anhydride, action on gallic acid, G. A. Prochazka, 296
Picraminic acid, cyanogen on, 264
Picric acid from morphine; 76
Pilocarpin, 78
Pilocarpin, action of nitric and hydrochloric acidon, 236
Piperidine, 262
Platinum, absorption of gases by, 239
Polarization, rotary, of chemical substances, 257
Polarization, rotation in the plane of, 173
Potash, alcoholic, on heptylene bromide, F. P. Venable, 254
Potash manufacture, evaporating mother lyes (Pat.), 43
Potassium almalgam, experiments on, 224
Potassium carbonate (Pat.), 45
Potassium, cyanide action on trichloracetate, 231
Potassium magnesium sulphate, from Kainit (Pat.), 43
Precipitates, separation of liquids from (Pat.), 39
Preservative for organic matters (Pat.), 109
Proceedings—January 6th, 1
February 3d, 10
March 3d, 24
April 7th, 34
May 5th, 57
May, adj. meeting 19th, 87
June 3d, 92
October 6th, 211
November 3d, 243
December 8th, 290
December, adj. meeting 15th, 291
Pyridic bases, etc., ethylenic chlorhydrin on, 272
Pyridin and quinolein, bromine on, 242
Pyridin, nicotinic acid from, 176
Pyrogallol, oxidation of, etc., 237
Pyrosulfuryl chloride, sp. gr. of vapor, 78
Pyrrhotite, analysis of, J. H. Stebbins, 214

Q

Quercitrin, etc., identity of coloring matter with, 206
Quinine, synthesis of, 236
Quinolein and pyridin, bromine on, 242
Quinolein, etc., ethylenic chlorhydrin on, 272
Quinolein and lutidin, 272
Quinones, determining constitution of, 199

R

Reducing properties of living cells 176
Resin oil, capronic acid in, 228
Resin spirit, some constituents of, 200
Resorcin, azoderivatives of, coloring matter from (Pat.), 42
Resorcin dyes, 268
Rhabdophane, a new British mineral, 203
Rosanilins, homologous and isomeric, 237

S

Salicylic acid, antiseptic properties, 238
Salts, crystallization from solutions of, 261
Salts, double, formed by fusion, 239
Salt, evaporating pans, etc. (Pat.), 79
Salt, forming into balls (Pat.), 39
Salt, Rock, from Saltville, Va., T Radcliffe, 255
Samarskite, earth-metals in, 222
Savory, essential oil of, 77
Sebacic acid, formation by distillation, 233
Sebacylic acid, bromo-substitution products, 204
Silver and lead from ores (Pat.), 43
Silver nitrate, oxychlorides of sulphur on, 223
Silver nitrate, thiophosphoryl chloride on, 224
Silver ores, reduction of, 241
Silver salts, etc., decompositions of, 235
Silicium, 240
Silicium, carbon disulphide on, 240
Silicium and carbon, new compounds of, 237

Silico-Molybdic acid, 77
Sizing (Pat.), 109
Soap boilers' waters, utilizing (Pat.), 109
Soap from animal and vegetable fats (Pat.), 111
Soda, ammonia, manufacture (Pat.), 43
Soda silicate, manufacture of (Pat.), 40
Sodic carbonate, precipitation of alums by, 258
Sodium bicarbonate, calcining (Pat.), 39, 110
Sodium bicarbonate, Manufacture of (Pat), 38
Sodium hydrate etc., action on feldspars etc., 199
Soils, Determination of Nitric acid in, 258
Spectra of carbon compounds, 144
Spectra Ultra Violet of Elementary Bodies, Photographs, 169
Spectroscopic study of chlorophyll, 257
Spectrum of Terbium, 223
Steel, Effects of compression on hardness of, 235
Strontum saccharate from molasses etc., (Pat) 108
Suberic acid, Formation by distillation, 233
Succinic acid, propyl and isopropyl, 270
Succinic acid, from tartaric by fermentation, 205
Sugar, grape, crystallized anhydrous A. Behr, 11
Sulpho carbanilid derivatives, 230
Sulpho cyanic acid etc., heat of formation etc., 234
Sulphonic compounds etc., chlorine on, 263
Sulpho group, expulsion by bromine, 175
Sulpho salicylic acid for colors (Pat), 80
Sulphur and Br, oombination with C., 226
Sulphur, new oxychloride of, 231
Sulphur oxychlorides on silver nitrate, 223
Sulphuretted hydrogen, direct estimation in gas, A. P. Hallock, 177
Sulphuric acid, (Pat), 45, 47
Sulphuric acid, nitrogen tetroxide with etc., 265
Sulphurous acid, utilizing, (Pat), 44
Sulphurous acid in wine, 263
Sunlight, reduction of metallic oxides in, A. R. Leeds, 3

T

Tannic acid, estimation of, N. H. Darton, 4
Tannic acid in wine, determination, 233
Tannic acid, value of methods for determining, N. H. Darton, 49, 62
Tanning hides (Pat), 45, 48
Tartaric acid, Dry products of distillation, 263
Tartaric acid, Manufacture of, L. H. Friedburg, 292
Tartaric acid, Succinic acid from, by fermentation, 205
Temperatures critical, 264
Terbium spectrum of, 223
Terebenthenic acid 227
Terpenes, molecular power of refraction of, 173
Tetra brom benzol, 175
Test paper, methyl violet, T O'C Sloane, 31
Theobromin etc., from xanthin, 264
Thermometer, mercurial with electrical alarm (Pat), 39
Thioformanilide, action of heat on, 206
Thiophosphoryl chloride on silver nitrate, 224
Thymol, synthesis from cuninol, 205
Tin, chlorosulphurous acid with, 261
Tin, protoxide, Alkaline solutions on, 235
Tolyl-methyl keton, 206
Tribromanilin, nitric acid on, 265
Tropin, 226, 264
Tungsten bronzes, 269

U

Ureas, sulphuretted from carbon oxysulphide, 236
Urea, transformation into cyanamide 221
Urine, large crystals of ammon. mag phos. in, 174
Uruguay, products from, 237

V

Vanadium, extraction from slags, etc., 241
Vapors, foul, apparatus for carrying off, (Pat.), 41
Vapor gauge, (Pat.), 45
Vesuvius ashes, composition of, 237

W

Water Buxton Thermal, examination of, 170
Water from the Isthmus of Panama, 242
Water Supply of the city of New York. E. Waller, 15
Water Supply of New York, contamination of, A. R. Leeds, 127
Waters Waste, disinfection of, (Pat.), 109
Weighing Flask to deliver as a burette, N. H. Darton, 6
Wine, Det. of astringent matters in, 242
Wines, champagne, clarification of, 234
Wine, glycol in, 202
Wine, sulphurous acid in, 205, 263
Wollastonite, action of sodium hyd. rate on, etc., 199

X

Xanthin, transformation into Theobromin, etc., 264

Z

Zinc alloys explosible, etc., 240
Zinc, extraction from ores, (Pat.); 79
Zinc oxide, deposit in a blast furnace, T. Radcliffe, 256
Zinc oxychloride, 239
Zinc as Pyrophosphate, determination of, G. C. Stone, 26
Zinc White, manufacture of, (Pa.), 111

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CONTENTS:

	Page.
Proceedings. December meeting	259
I. A Chemical, Biological and Experimental inquiry into the present and proposed future water supply of the City of Albany; By Dr. Albert R. Leeds	261
Abstracts from Chemical Journals:	
General and Inorganic Chemistry	283
Organic Chemistry	288
Analytical Chemistry	290
Abstracts of American Patents relating to Chemistry	297
Errata, Volume VII	302
Index " "	
Title page " "	
Officers for 1886	
List of Members	

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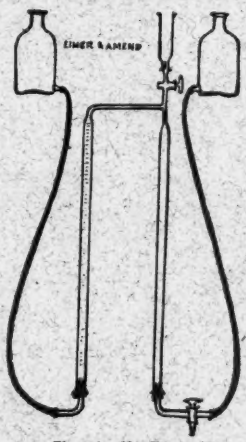

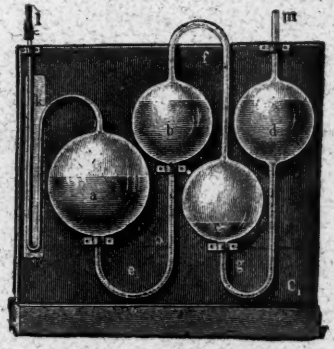

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